EFFECT OF SURFACE CONDITIONING METHODS ON REPAIR BOND STRENGTH OF MICROHYBRID RESIN MATRIX COMPOSITE

by

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INTRODUCTION

Resin matrix composite is one of the most common materials used for restoring teeth at the present time. The mechanical properties and esthetics of this material have made it preferable for many practitioners. However, this material can develop defects as a result of wear, fracture, or discoloration after a long period of service. Color mismatch can also generate patient dissatisfaction requiring replacement of the restoration. The replacement of resin matrix composite is a time-consuming and may result in weakening of the tooth as additional structure is removed.

Alternative treatment, such as selective repair, is a conservative option and can increase the longevity of restorations at a lower cost than replacement.[1-3]

In general, bonding between two resin composite layers is satisfactorily achieved if there is an unpolymerized layer remaining with carbon-carbon double bonds. However, it is more difficult to bond to a polished, abraded, or contaminated layer due to the absence of any unreacted carbon-carbon double bond.

Several techniques have been proposed to improve bonding between old and new resin matrix composite layers. There have been attempts to use mechanical surface treatments, such as bur or disc abrasion, micromechanical surface treatments with airborne aluminum oxide particles; chemical surface treatments utilizing tribochemical silica coating, hydrofluoric acid, phosphoric acid, and silane; and bonding agents to condition the old resin composite surface prior to bonding a new layer. However, little is known of their efficacy.

This study compared several common surface treatments to determine their level of efficacy to improve the bond strength between old and new resin composite layers.

REVIEW OF LITERATURE

Resin matrix composite has been used widely for both direct and indirect restorations. Development began in 1955 when Buonocore[4] introduced orthophosphoric acid etching to improve adhesion of acrylic resin to enamel. In 1962, Bowen[5] made significant improvements in tooth-color materials with the development of Bis-GMA monomer, which increased the number of cross-linking reactions and improved the physical properties of acrylic resin. Basically, there are three major components in resin matrix composite; the resin matrix (organic phase), inorganic fillers (dispersed phase), and a coupling agent (interfacial phase). Resin matrix composites in use can be classified by the size, loading, and distribution of filler particles into hybrid, microfilled, microhybrid and, the newest, nanohybrid composites. Microfilled resin matrix composite provides polishability with improved esthetics due to very small filler particles ranging from 0.04 μm to 0.4 μm (20 to 50 volume percent).[6] Hybrid resin matrix composite contains inorganic filler particles ranging from 0.04 μm to 4 μm that improve physical properties and provide acceptable polishability.[6] With modern technology, small and submicron fillers can be blended into microhybrids (0.04 μm to 1 μm), and nanohybrids (0.005 μm to 0.01 μm) that have enough strength and esthetics to be used for posterior areas and anterior areas, respectively.[6]

The use of resin matrix composites in high stress-bearing areas often results in both direct and indirect restorations deteriorating due to fracture, leakage, and secondary decay.[7, 8] The average longevity of resin matrix composites is approximately seven to eight years,[9,10] and the two most frequently cited reasons for restoration replacement are

secondary caries (50 percent) and fracture (25 percent).[10] Replacement of resin matrix composite restorations has become a common restorative procedure.[11] When resin matrix composites are replaced, some sound tooth structure must be removed to gain access to an etchable substrate. This cycle of placement and replacement is often accompanied by weakening of the tooth.[8] Tobi et al.[12] have shown that replacement of resin matrix composite restorations have a lower cost-effectiveness than replacement of amalgam restorations.

The treatment options to replacement are refurbishing, sealing of defective margins, and repair. When the defects are of moderate size, they can be treated with refurbishing, for example, refinishing or polishing of the margins. Sealing of defective margins with pit and fissure sealant or flowable resin materials can be done to non-carious margins. Bulk fracture involving the loss of a portion of a restoration should be analyzed for the cause of the fracture before repair is contemplated.[10]

Gordan and colleagues[1] evaluated replaced and repaired restorations and found that within two years, the repairing of defective and stained margins showed good results according to Modified Ryge USPHS Clinical Criteria.[13] They concluded that repair should be considered before replacement. Moncada et al.[1-3] also evaluated optional treatments at 12 months, 24 months, and 36 months and found an improvement of USPHS/Ryge scores for the sealing, repairing, and refurbishing treatments compared with untreated subjects.

Bond strengths of interfacial surfaces between repaired and aged resin matrix composites have been reported in many studies. As early as 1975, Causton[14] prepared surfaces of eight-week aged resin matrix composites with 600-grit emery paper and

concluded that the bond strengths were clinically acceptable, although they were significantly lower than the intact cohesive strength of resin matrix composites. Chan and Boyer[15] found that bond strengths of repaired resin matrix composites with microfilled resins were around 76.4 percent of their intact strengths compared with 67.7 percent of conventional resins. In 1984, Boyer et al.[16] began testing light-cured resin matrix composites with different storage times and found that bond strengths were decreased with time to approximately 27 percent of cohesive strength without bonding agent and 48 percent with bonding agent after 24 hours. There was no significant decrease in repaired bond strength after 24 hours,[17] and different types of resin matrix composites did not show any differences or advantages in repaired bond strength.[18] When resin matrix composite was ground or cut, inorganic filler particles were exposed, which were unlikely to bond with the new resin matrix composite.[16] Repair of resin matrix composite with chemically identical matrices did not provide greater bond strength than those with a different matricies.[17-19] Instead, the availability of carbon-carbon double bonds in the originally cured polymer reacted with the repair resin matrix composite allowing the materials to adhere to each other.[18, 19] Other studies compared the effect of the presence and absence of an oxygen inhibition layer. The conclusion was that an oxygen-inhibition layer was not necessary for additional resin coupling, at least up to 14 days after polymerization.[20] However, the ability to bond to a new resin matrix composite layer depended on the presence of unreacted carbon-carbon double bond within the cured resin matrix composite.[21] Shen et al.[22] reported that undercuts made by a bur in aged resin matrix composite surfaces did not improve the strength of repaired resins. However, temperature may influence bonding ability. Papacchini et al.[23] found

that composites preheated to 37 °C provided greater bond strength than at 4°C or 23°C because resin viscosity is temperature-dependent and directly relates to the handling characteristics of the composites.

Theoretically, two factors that impair adhesive ability are the low chemical bonding potential of the aged substrate, and the incomplete penetration of the highly viscous fresh resin matrix composite into surface pits and depressions.[22, 24] Due to the limited amount of unreacted carbon-carbon double bond available for reacting with new resin on the old and contaminated resin matrix composite, some investigations have focused on surface treatments with mechanical and chemical techniques other than basic abrading with a bur[17, 20, 24-30] or disc to improve bond strength of repaired resin matrix composites. *In vitro* studies have examined airborne particle abrasion with 30 μm to 50 μm aluminum oxide,[20, 24, 27, 29-38] tribochemical silica coating,[27, 29, 35, 39-42] 9.6-percent hydrofluoric acid etching (HF),[20,26,30,31,34,35,37-39, 41] phosphoric acid etching,[24, 28, 30, 41] silanization,[24, 26, 28-30, 38, 43-45] and application of bonding agent.[17, 23, 25-27, 30, 33, 36, 40, 42-44, 46, 47] However, the results of these studies were inconclusive and did not indicate a method that routinely produced the greatest interfacial bond strength.

Airborne particle abrasion with aluminum oxide particles enhances the ability of new resin matrix composite to interlock mechanically into the aged substrate by providing increased surface area for micro-mechanical bonding. A number of studies showed the highest interfacial bond strength with this method when compared with other techniques.[20, 25, 30-33, 35, 38] It has been reported that this surface conditioning, along with the application of a bonding agent, could result in a repaired bond strength that was almost identical to the cohesive strength of the original resin matrix composite.[25, 36]

Silica coating utilizes air abrasion with 30 μm Al_2O_3 particles modified with silica (CoJet).[41] The coating increases the surface energy of aged composite and embeds a proprietary silicate ceramic layer of submicron particles that combine with silanization to provide a chemical and micromechanical bond for the repaired resin matrix composite. Some studies showed equivalent[35] or superior results to air abrasion.[29] However, Rathke et al.[27] found that CoJet Sand with silanization was not superior to bur or air abrasion when used with an adhesive agent. Moreover, if exposed enamel or dentin were involved, the use of silica coating and silanization would interfere with the resin matrix composite bond to enamel or dentin at repaired sites.

Etching with hydrofluoric acid also improved bond strength of repaired resin matrix composite by selectively removing inorganic fillers to create porosities. Successful results were found in a study by Trajtenberg and Powers[34] that showed the highest bond strength of repaired indirect resin matrix composite occurred with 8-percent hydrofluoric acid etching for 15 seconds followed by the application of a resin/silane primer. Furthermore, another study by the same authors reported that there were no differences in the repaired bond strengths of indirect composite when using 5-, 8-, or 9.5-percent concentrations of hydrofluoric acid at 5 s, 10 s, 15 s, 30 s, or 60 s of etching time.[48] Conversely, Papacchini et al.[20] showed lower bond strengths with this method compared with other mechanical methods. Some studies have reported that 30-s to 60-s applications of hydrofluoric acid caused softening and porosity of the resin matrix composite surface and reduced the bond strength of repaired indirect composites.[30, 31, 35] In addition, some resin matrix composites containing non-etchable quartz instead of

glass, or microfilled resin matrix composites containing SiO_2 may not be affected by this acid.[31]

Phosphoric acid etching is much safer to use clinically than the stronger hydrofluoric acid. However, it has been shown to be ineffective in creating any micromechanical retention in resin matrix composite surfaces.[20, 24, 28, 30, 41] Instead, its action is primarily to clean and remove superficial debris and organic films.

Bonding agent was also found to improve repaired bond strength, because of its low viscosity, which enhances wettability and surface penetration.[17,26, 46] Brosh et al.[26] concluded that there were three potential mechanisms for improved bonding with low viscosity agents. These were chemical bonding to the resin matrix, chemical bonding to the exposed filler particles, and improved micromechanical retention due to penetration of the low viscosity resin into microcracks in the resin matrix. Oztas et al.[33] revealed that using bonding after air abrasion resulted in higher repair bond strength. Another study showed that multi-step adhesion primer yielded higher bond strengths than one-step primer,[44] and etch and rinse systems were better than self-etch systems.[49] However, these findings were in contrast to those of Cavalcanti et al.,[36] who found that using Clearfil SE (self-etch) containing 10-methacryloyloxydecyl dihydrogen phosphate provided greater repair bond strength after air abrasion than single bond (etch and rinse). Cavalcanti et al.[36] concluded that there was little or no microleakage of the interfacial layer when using a bonding agent as an intermediate regardless of the type of surface treatment.[50] Papacchini et al.[23, 47] found that flowable resin provides higher repair bond strength to sandblasted surfaces than regular unfilled resin.

A silane coupling agent is a molecule with two functional groups: a silanol group for reacting with inorganic filler particles, and an organo-functional group for reacting with the methacrylate in the resin matrix.[41] It has been used clinically for promoting a covalent bond between porcelain and resin for ceramic repairs and was shown to improve bond strength between resin matrix composites after HF application,[38] after bur abrasion,[26, 28, 43, 44] and after airborne particle abrasion[29, 30] due to the exposure of filler particles.[26, 29] However, other studies found that hydrofluoric acid reduced bond strength after bur abrasion,[24] and airborne particle abrasion.[38] Silanization seemed to be more effective with higher-filled than lower-filled resins.[29] Papacchini et al.[47] found that using air drying at 38°C after silane application following sandblasting significantly improved bond strength over the use of air drying at 23°C because heating may have promoted additional evaporation of residual water and solvents from the silane coupling agent, which resulted in enhanced bonding.

The erbium, chromium: yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser is the most recent system with surface preparation potential. This type of laser (wavelength 2.78 μm) can be effectively absorbed by water and also hydroxyapatite crystal. The absorption of photon energy causes vaporization resulting in macroscopic and microscopic irregularities via micro-explosions of the material surface. The Er,Cr:YSGG pulsed laser, using an air-water spray, can cut enamel, dentin, cementum, bone, and soft tissue efficiently without significant thermal side effects. This laser operates in a pulse mode, which achieves high energy up to 300 mJ with power up to 6W.[51] Some studies have reported that tooth surfaces irradiated by Er,Cr:YSGG at a 90-degree angle showed characteristic rough, and clean surface without debris.[52-54] In addition to soft and hard

tissue surgeries, erbium laser can be used to remove resin matrix composite. The previous version of erbium: yttrium-aluminum-garnet (Er:YAG) was studied for effectiveness of resin matrix composite removal.[55, 56] Er,Cr:YSGG also has the ability to remove resin matrix composites at approximately the same rate as dentin.[57] Er,Cr:YSGG are generally set at the following parameters:

- Pulse duration 140 μs.
- A repetition rate of 20 Hz.
- A power output range from 0.0 to 6.0 W.
- Pulse energy at 0 to 300 mJ per pulse.
- Energy density per pulse from 0.0 to 68.02 J per square centimeter.

Light delivery is through a fiber-optic system to a terminal sapphire tip with an adjustable air-water spray handpiece.[58] Recent studies have set the power output at 6.0 W with 90-percent air pressure and 75-percent water pressure for enamel etching, and 4.0 W with 65-percent air pressure and 55-percent water pressure for dentin etching.[54, 59, 60] The erbium laser was evaluated for surface treatment of indirect resin matrix composites in a study by Burnett et al.[61] where they found that surface preparation by the laser showed the highest tensile bond strength when compared with hydrofluoric acid etching or air abrasion.

Based on this evidence, the aims of this *in vitro* study are 1) To evaluate various surface treatments on shear bond strength of repair between aged and new microhybrid resin matrix composite, and 2) To assess the influence of silane coupling agent applied after different surface treatments.

NULL HYPOTHESES

1) There are no differences in shear bond strength of repaired resin matrix composite when aged resin composite surfaces are prepared with airborne particle abrasion with 50-µm aluminum oxide, tribochemical silica coating, Er,Cr:YSGG laser, or no surface preparation.

2) The application of silane coupling agent following surface preparation does not increase repaired bond strength.

ALTERNATIVE HYPOTHESES

1) Shear bond strength of repaired resin matrix composite increases when aged resin composite surfaces are prepared with airborne particle abrasion with 50-µm aluminum oxide, tribochemical silica coating, or Er,Cr:YSGG laser in comparison with no surface preparation.

2) The application of silane coupling agent following surface preparation increases bond strength after repair of a resin composite surface.

MATERIALS AND METHODS

SPECIMEN PREPARATION

Plastic molds 16 mm in diameter were poured with self-cure acrylic resin (Bosworth Fastray, Bosworth Co, IL). A hole was made in each mold approximately 8 mm in diameter and 2 mm in depth. Retention was created at the bottom of the hole using an inverted bur. Each acrylic surface was polished sequentially with 240-grit, 320-grit, 400-grit, and 600-grit SiC paper. Microhybrid resin matrix composite (Point 4, shade A4, SDS Kerr, Orange**,** CA) was placed into the mold using a composite dispenser gun. The top surface of each specimen was pressed perpendicular to the long axis of the cylinder with a Mylar strip and glass slide in order to obtain a flat surface and remove excess material. Only the glass slide was removed and specimens were cured with an LED visible light-curing unit (L.E. Demetron I, SDS Kerr) for 40 seconds. The light tip was kept perpendicular to and in contact with the Mylar strip surface to receive maximum curing depth. Light intensity was monitored before (> 900 mW/cm^2) and after (> 900 mW/cm^2) the experiment with a visible light-curing light meter (CURE RITE, Dentsply, York**,** PA). All specimens with resin composite on the top were polished with the same sequence of SiC paper to remove the excess of resin and to make the surface perpendicular to the specimen's long axis. Eighty specimens were made (Figure 1).

AGING METHOD

Aging was simulated on all specimens by thermocycling 5000 times between 6°C and 51°C (+/- 2°C). A 30-second dwell time and 10-second transfer time were used.

SURFACE CONDITIONING METHODS

All specimens were randomly assigned into three experimental groups and one control group.

Airborne Particle Abrasion (n = 20)

Air abrasion was performed with an intraoral air abrasion unit (Microetcher II, Danville Engineering, San Ramon**,** CA) using 50-µm aluminum oxide particles (Patterson, St. Paul**,** MN) (Figure 2) moving slowly in a circle to create a uniform irregularity at a distance approximately 5 mm perpendicular to the specimen surface for 10 s with an air pressure of 60 psi. Specimens were rinsed with water for 10 s, and dried with an air syringe for 5 s.

Standardization for distance between the tip and the specimen was made using stone as a base for securing the specimen, and by using a plastic cone with resin matrix composite as a parallel guide on the top. An analyzing rod and surveyor were used to ensure a perpendicular approach to the specimen surface (Figure 3, 4).

Tribochemical Silica Coating (n = 20)

Silica coating was performed with the same intraoral air abrasion unit using 30-µm Al_2O_3 particles that are modified with silica (CoJet System, 3M, St. Paul**,** MN) moving slowly in a circle to create a uniform irregularity at a distance of approximately 5 mm perpendicular to the specimen surface for 10 s with an air pressure of 60 psi. Specimens were rinsed with water for 10 s, and dried with an air syringe for 5 s.

Standardization was the same as for airborne particle abrasion (Figure 3, 4).

Er,Cr:YSGG Laser (n = 20)

Laser preparation was performed with an Er,Cr:YSGG laser device (Waterlase, Biolase Technology, San Clemente, CA) in noncontact and focused mode. The specimen and the jig were moved slowly in a circle at a distance of approximately 1 mm from the cylinder fiber tip for 10 s. The handpiece was adjusted so that the fiber tip irradiated at 1 mm perpendicular to the specimen surface and was secured in silicone connected to a stone jig (Figure 5). The laser parameters were set at a wavelength of 2.78 μm, a pulse frequency of 20 Hz, a pulse duration of 140 μs, a power of 4.0 w, an air pressure setting of 65 percent, and a water pressure setting of 55 percent (Figure 6).

Control (n = 20)

No surface treatment was performed on the control group.

SURFACE CLEANING, SILANIZATION AND ADHESIVE APPLICATION

After surface conditioning, all specimen surfaces were cleaned with 35-percent phosphoric acid (Ultra-Etch, Ultradent Products, South Jordan**,** UT). Acid gel was left on the resin surface for 15 s; then it was rinsed with water for 10 s, and dried with air for 5 s. Each group was assigned into two subgroups (n = 10): a) No silanization and b) With silanization (Figure 7). The silane coupling agent (Silane, Ultradent Products) was applied to the subgroup b specimens and air dried after 60 s.

An adhesive agent (OptiBond Solo Plus, SDS Kerr) was applied with a microbrush on the surface of all specimens (two consecutive layers). The solvent was thinned out using canned compressed air and then light-cured for 20 seconds.

GROUPS

The specimens were placed in the following groups (Figure 7):

Group 1) Airborne particle abrasion with 50-µm aluminum oxide without silanization.

Group 2) Tribochemical silica coating without silanization.

Group 3) Er,Cr:YSGG without silanization.

Group 4) Control without silanization.

Group 5) Airborne particle abrasion with 50-µm aluminum oxide with silanization.

Group 6) Tribochemical silica coating with silanization.

Group 7) Er,Cr:YSGG with silanization.

Group 8) Control with silanization.

APPLICATION OF THE RESIN MATRIX COMPOSITE REPAIR

A new layer of resin matrix composite was applied to the treated surfaces using a plastic mold to create cylinders of approximately 4 mm diameter and 2 mm high. These were then light-cured for 40 seconds. After repair, the specimens were stored in distilled water at 37°C for 24 hours.

SHEAR BOND STRENGTH TEST

After measuring diameters with a caliper (Figure 8) and recording, all specimens were secured in a special holder in a universal testing machine (Sintech Renew 1123, MTS Systems Corp.**,** Eden Prairie, MN) (Figure 9). The direction of force was perpendicular to the long axis of specimens. A stainless steel ring with an inner circular

knife-edge was adapted at the interface of the aged and new resin matrix composite surface (Figure 10). Shear load was applied with a crosshead speed of 1 mm/min until fracture. The force was calculated in megapascals (MPa).

TYPE OF FAILURE ANALYSIS

All specimens and broken parts were investigated for type of failure under a stereomicroscope (Prior, Rockland, MA) at X10 and X15.

SURFACE ROUGHNESS EVALUATION

Twelve additional resin composite specimens were prepared and aged by the same method. They were assigned into three surface treatment groups and one control group (n = 3). After surface treatment, all specimens were cleaned with 35-percent phosphoric acid, rinsed, and dried. Surface roughness was determined with a profilometer (Surtronic 3^{+}, Taylor Hobson USA, West Chicago, IL) operated with Taly Profile Lite software (Taylor Hobson USA). Each specimen was measure six times (4 mm distance) in six different locations. The average Ra value from each specimen was recorded.

SCANNING ELECTRON MICROSCOPE (SEM) EVALUATION

Specimens used from the surface roughness experiment were mounted on a specimen holder with electrically conductive double-sided sticky tape and observed with a scanning electron microscope (model JSM-5310LV; JEOL, Peabody, MA). The SEM was operated with an accelerating voltage of 10 kV using both secondary and

backscattered electron detectors for imaging. Micrographs were taken at magnification X750 in order to document the surface texture created by the different surface treatment.

STATISTICAL ANALYSIS

A two-way analysis of variance (ANOVA) model was used to assess the impact of surface conditioning (4 levels), silanization (yes or no), and their interaction on peak stress. Pair-wise comparisons were adjusted for multiple comparisons using Tukey's multiple comparisons procedure to control the overall significance level at 5 percent.

Mantel-Haenszel chi-square tests for ordered categorical responses were used to compare the groups for differences in failure mode. Pair-wise comparisons were adjusted for multiple testing using the Sidak method. P-values are presented with and without multiple comparisons adjustment.

RESULTS

SHEAR BOND STRENGTH TEST

The interaction between conditioning and silanization was significant ($p = 0.0163$), indicating that comparisons of silanization must be evaluated for each conditioning method and comparisons of conditioning methods must be evaluated separately with and without silanization. Airborne particle abrasion had significantly higher peak stress than Er,Cr:YSGG laser without silanization ($p < 0.0001$) and with silanization ($p = 0.0002$) and higher peak stress than Control without silanization ($p < 0.00001$) and with silanization ($p < 0.00001$). Airborne particle abrasion did not have significantly different peak stress than Tribosilica coating without silanization ($p = 0.70$) or with silanization ($p = 0.33$). Tribosilica coating had significantly higher peak stress than Er,Cr:YSGG laser without silanization ($p < 0.0001$) and with silanization ($p < 0.0001$) and significantly higher peak stress than control without silanization ($p < 0.0001$) but not with silanization ($p = 0.16$). Er,Cr:YSGG laser and control did not have significantly different peak stress without silanization ($p = 1.00$) or with silanization ($p = 0.11$). There was no effect of silanization on peak stress overall ($p = 0.34$) for any of the surface conditioning methods ($p = 0.76$ for airborne particle abrasion, $p = 0.39$ for tribosilica coating, $p = 1.00$ for Er,Cr:YSGG laser, $p = 0.39$ for control).

Therefore, the first null hypothesis was rejected and the second null hypothesis was supported.

TYPE OF FAILURE

Type of failure was categorized into adhesive (Figures 11 and 12), mix (Figures 13 and 14), and cohesive failure (Figures 15 and 16). Airborne particle abrasion without and with silanization (group 1) had significantly higher cohesive failure than Er,Cr:YSGG laser without silanization ($p = 0.0111$) and with silanization ($p = 0.0139$) (Table I, and II). Airborne particle abrasion did not have significantly different cohesive failure than tribosilica coating without silanization ($p = 1.00$) or with silanization ($p = 0.99$) and did not have significantly different cohesive failure than control without silanization ($p = 0.86$) or with silanization ($p = 1.00$). Tribosilica coating without silanization had significantly higher cohesive failure than Er,Cr:YSGG laser without silanization ($p = 0.0111$) or with silanization ($p = 0.0139$). Tribosilica coating without silanization did not have significantly different cohesive failure than tribosilica coating with silanization ($p = 0.99$) and did not have significantly different cohesive failure than control without silanization ($p = 0.86$) or with silanization ($p = 1.00$). Tribosilica coating with silanization had marginally higher cohesive failure than Er,Cr:YSGG laser without silanization ($p = 0.06$) and with silanization ($p = 0.06$). Tribosilica coating with silanization did not have significantly different cohesive failure than control without silanization ($p = 1.00$) or with silanization ($p = 0.99$). Er,Cr:YSGG without silanization had significantly lower cohesive failure than control with silanization ($p = 0.0111$). Er,Cr:YSGG without silanization did not have significantly different cohesive failure than Er,Cr:YSGG with silanization ($p = 1.00$) or control without silanization ($p = 0.13$). Er,Cr:YSGG with silanization had significantly lower cohesive failure than control with silanization ($p = 0.0139$).

SURFACE ROUGHNESS

Surface roughness was analyzed after a log-transformation due to the assumptions required for the ANOVA. All four surface treatments had significantly different surface roughness: Er,Cr:YSGG > airborne particle abrasion > tribosilica coating > control. P-values for the comparisons were all 0.0001 except for tribosilica coating vs. control which had a p-value of 0.0019.

SEM EVALUATION

SEM examination of surface treated resin composite revealed different textures. Airborne particle abrasion with 50 μm aluminum oxide particles produced a roughened, highly irregular surface with resin composite asperities created among numerous micro-retentive fissures (Figures 17 and 18). Tribochemical silica coating also created a roughened and irregular surface but less than airborne particle abrasion (Figures 19 and 20). Er,Cr:YSGG created the roughened and macro-irregularities on resin composite surface (Figure 21). Also, surface treatment with Er,Cr:YSGG created cracked lines on the resin composite surface (Figure 22). Resin composite surface without any surface treatment showed a uniformly smooth on the resin composite surface (Figures 23 and 24).

TABLES AND FIGURES

TABLE I

Type of failure

Group No.	Cohesive failure	Mixed failure	Adhesive failure
1	10	-	-
2	10	-	-
3	1	6	3
4	7	3	-
5	10	-	-
6	8	2	-
7	1	4	4
8	10	-	-

*Note: One specimen was lost during the test on group 7.

TABLE II

Statistical comparison of type of failure

Comparison	unadjusted p-value	adjusted p-value
1 vs 2	1.00	1.00
1 vs 3	0.0004	0.0111*
1 vs 4	0.07	0.86
1 vs 5	1.00	1.00
1 vs 6	0.15	0.99
1 vs 7	0.0005	0.0139*
1 vs 8	1.00	1.00
2 vs 3	0.0004	0.0111*
2 vs 4	0.07	0.86
2 vs 5	1.00	1.00
2 vs 6	0.15	0.99
2 vs 7	0.0005	0.0139*
2 vs 8	1.00	1.00
3 vs 4	0.0050	0.13
3 vs 5	0.0004	0.0111*
3 vs 6	0.0023	0.06*
3 vs 7	0.66	1.00
3 vs 8	0.0004	0.0111*
4 vs 5	0.07	0.86
4 vs 6	0.61	1.00
4 vs 7	0.0043	0.11
4 vs 8	0.07	0.86
5 vs 6	0.15	0.99
5 vs 7	0.0005	0.0139*
5 vs 8	1.00	1.00
6 vs 7	0.0022	0.06*
6 vs 8	0.15	0.99
7 vs 8	0.0005	0.0139*

* = significant difference.

TABLE III

Surface roughness (Ra value)

Surface treatment	Airborne particle abrasion with 50µm Al_2O_3 particle			Tribochemical silica coating			Er,Cr:YSGG			Control		
Specimen number	1	2	3	1	2	3	1	2	3	1	2	3
Ra Mean (µm)	2.34	2.19	2.28	1.60	1.59	1.55	14.77	14.25	12.93	0.29	0.43	0.24
S.D.	0.22	0.07	0.26	0.06	0.11	0.16	1.95	2.32	2.05	0.05	0.03	0.06
Average Ra	2.27 µm (+/- 0.07)			1.58 µm (+/- 0.03)			13.98 µm (+/- 0.95)			0.32 µm (+/- 0.1)		

FIGURE 1. Image of specimen preparation before surface conditioning.

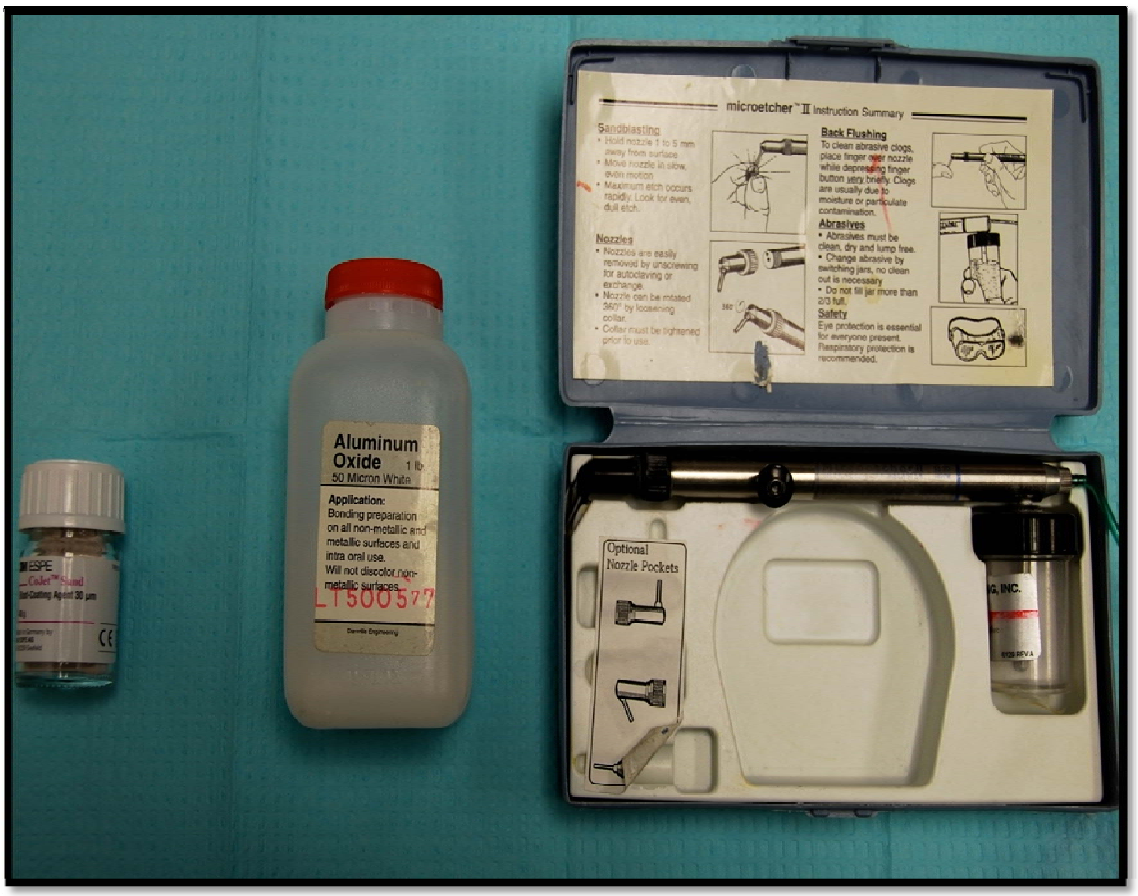

FIGURE 2. Image of an intraoral air abrasion unit.

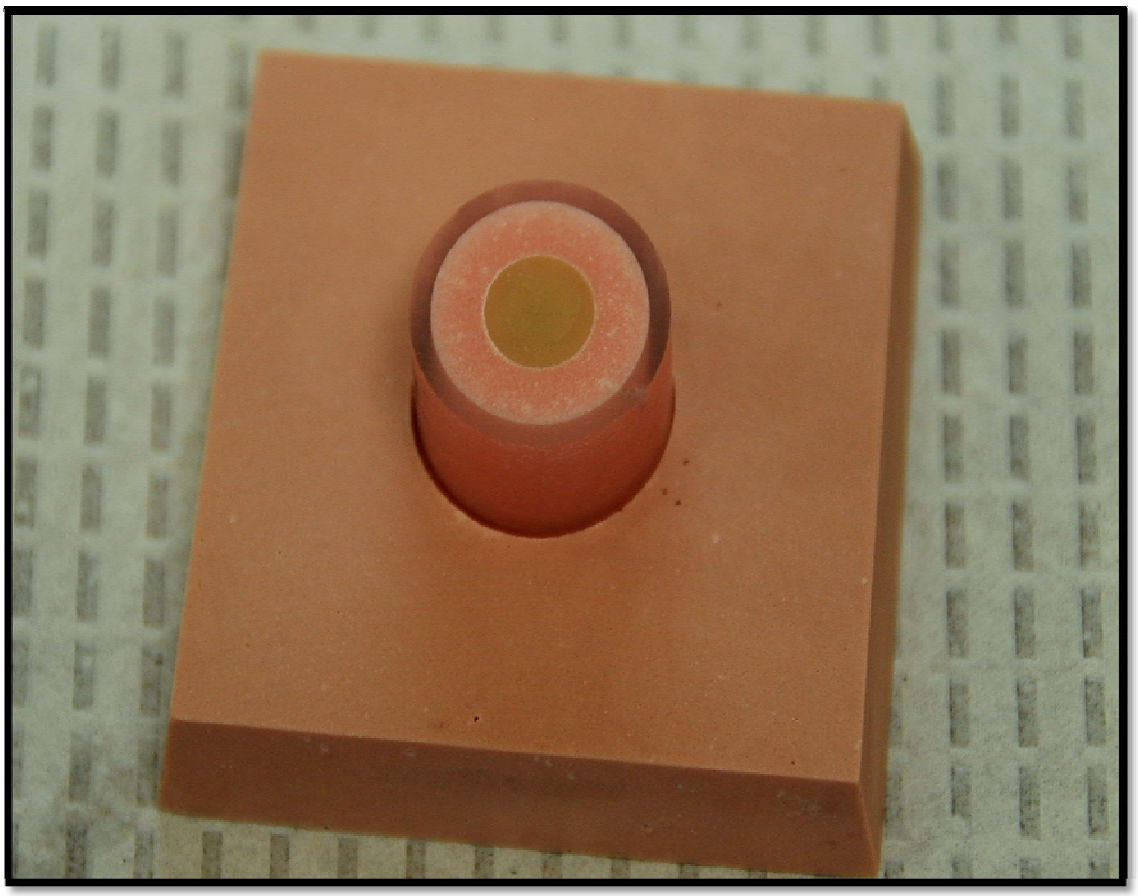

FIGURE 3. Image of specimen in the stone base.

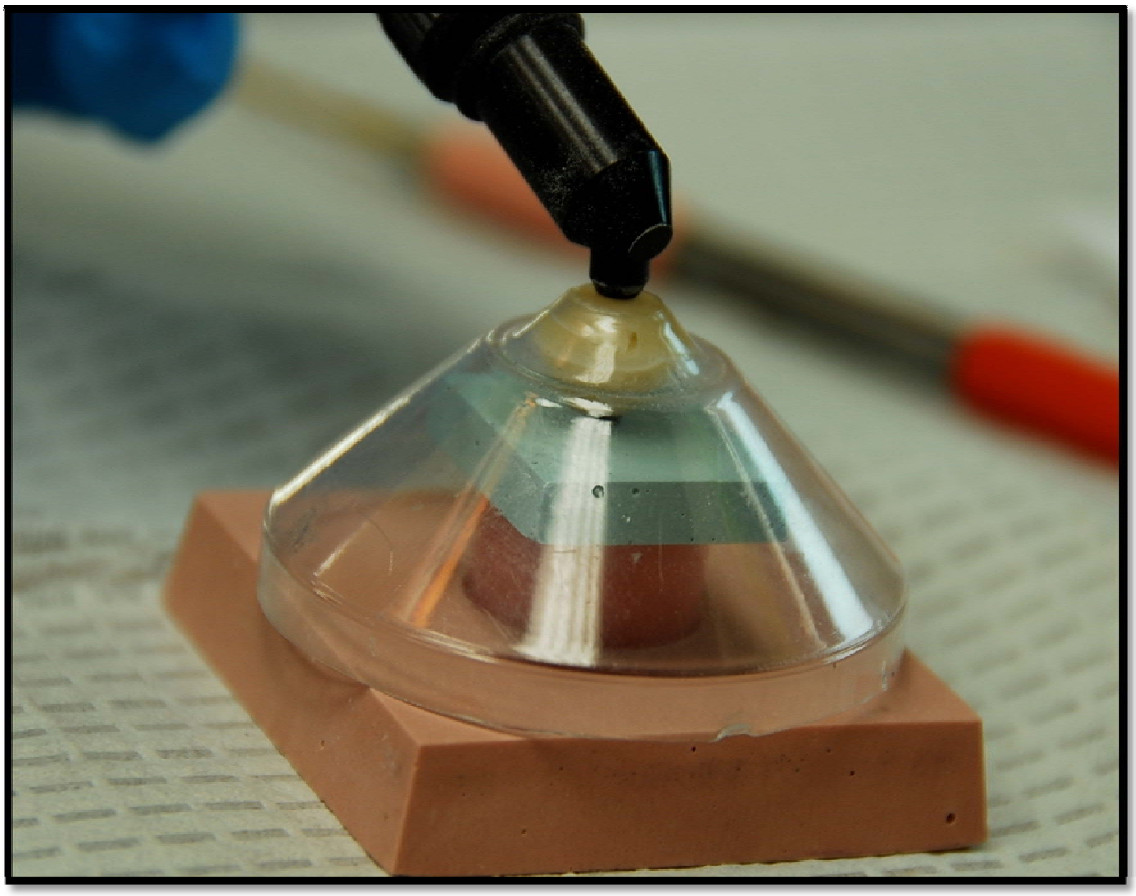

FIGURE 4. Image of standardization device for an intraoral air abrasion tip.

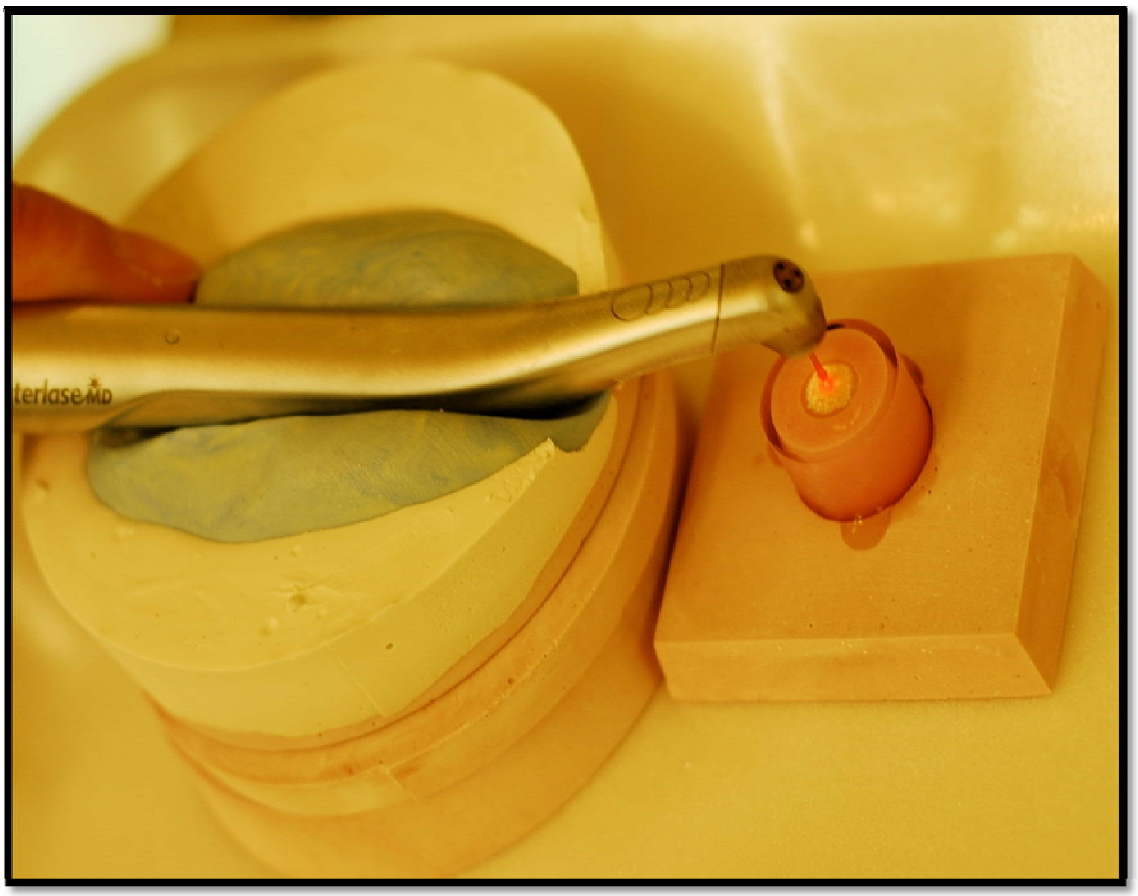

FIGURE 5. Image of standardization device for laser tip.

FIGURE 6. Image of Er,Cr:YSGG laser unit with the parameter setting.

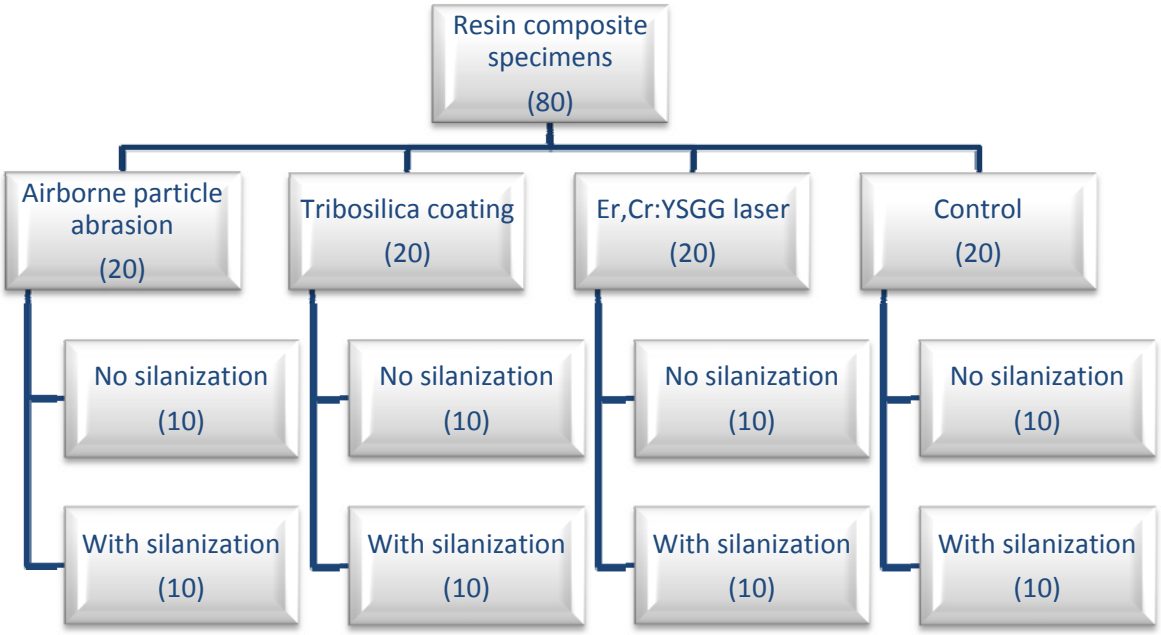

FIGURE 7. Flowchart showing specimen assignment.

FIGURE 8. Image of caliper for measuring dimension of specimens before loading.

FIGURE 9. Image of a universal testing machine.

FIGURE 10. Image of specimen secured before shearing.

FIGURE 11. Image of adhesive failure 1.

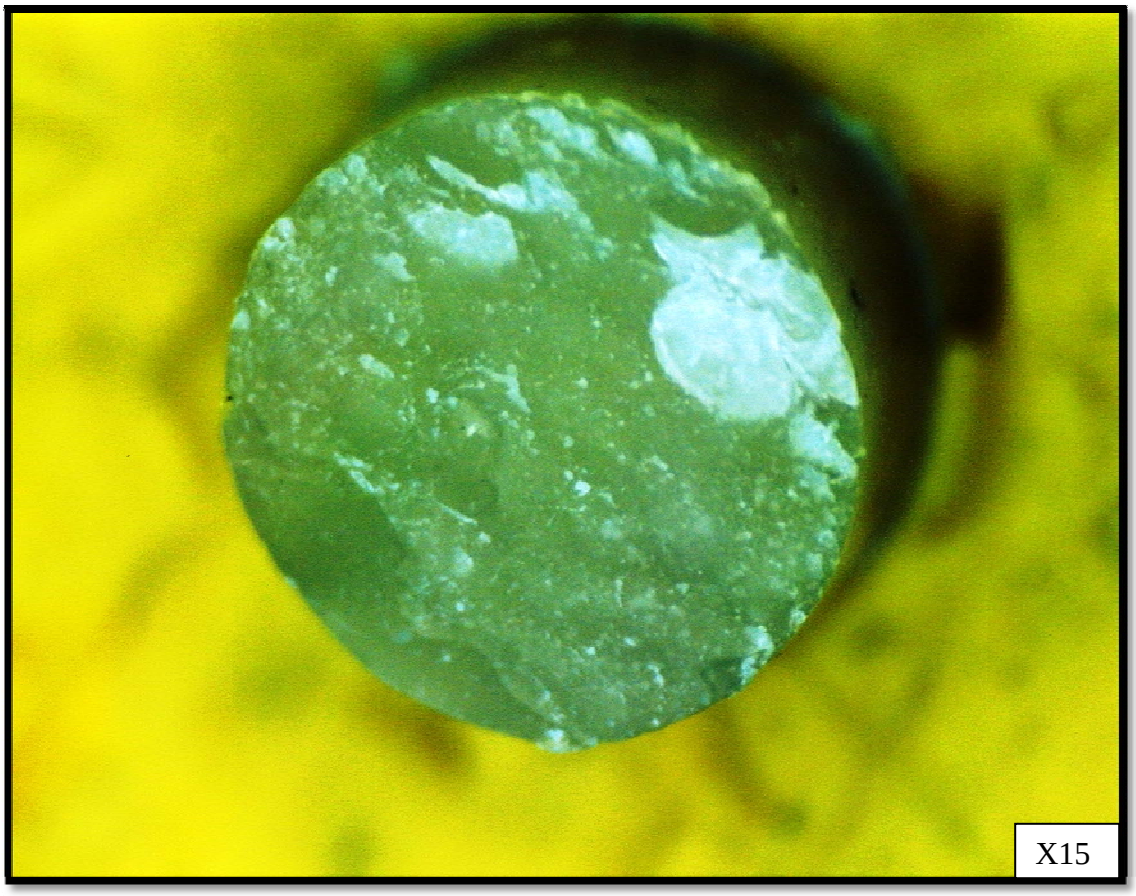

FIGURE 12. Image of adhesive failure 2.

FIGURE 13. Image of mix failure 1.

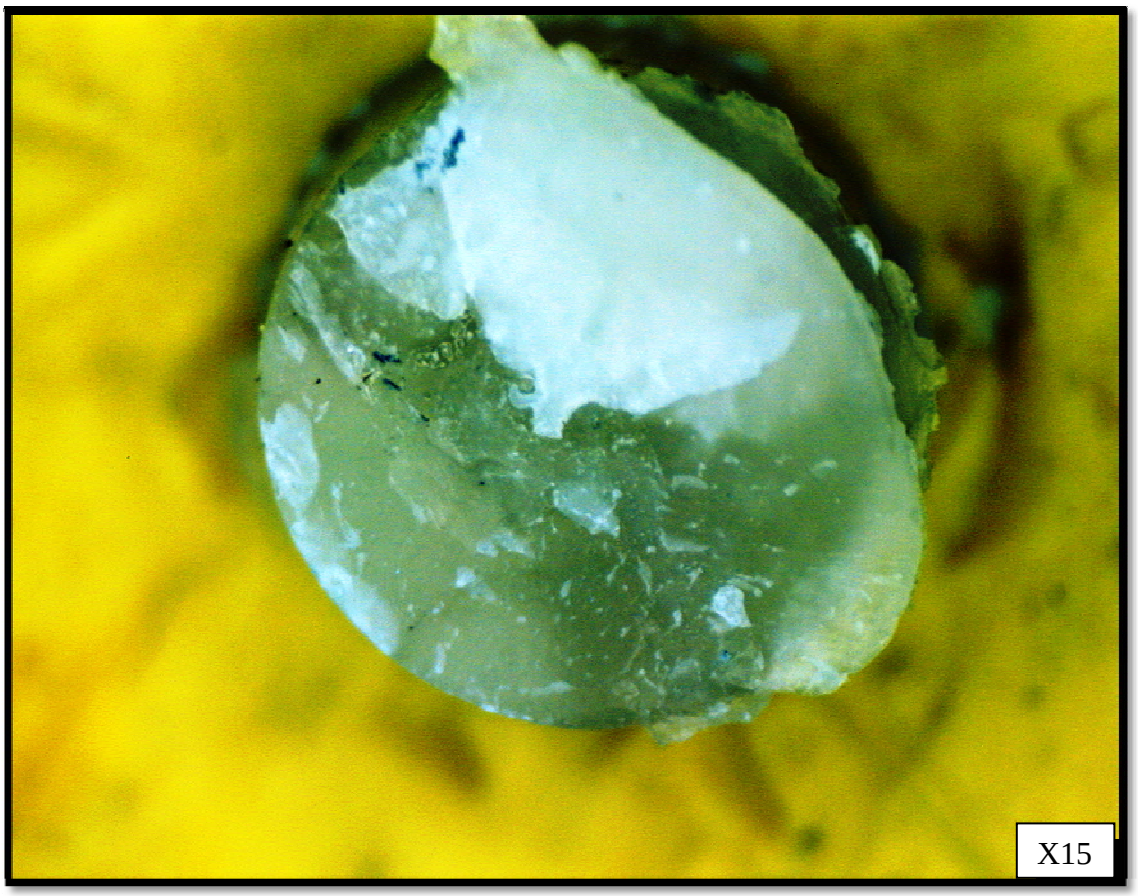

FIGURE 14. Image of mix failure 2.

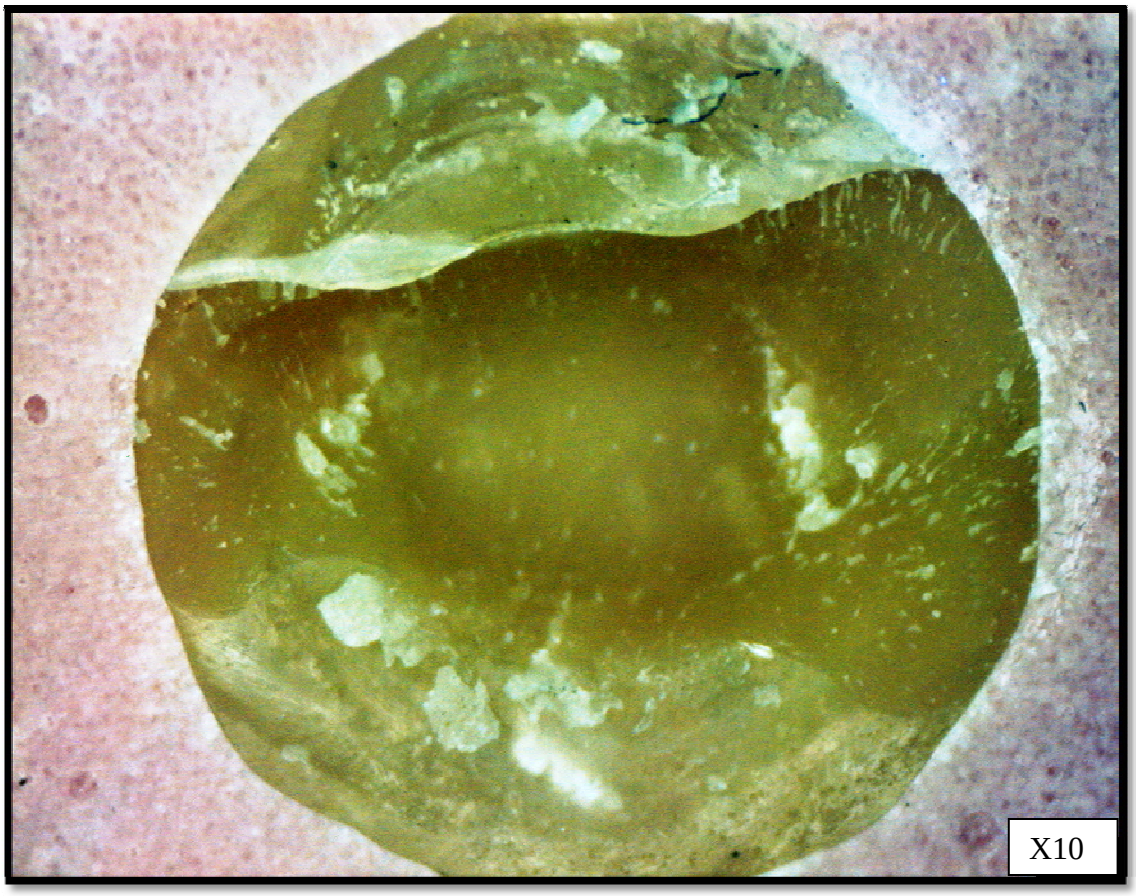

FIGURE 15. Image of cohesive failure 1.

FIGURE 16. Image of cohesive failure 2.

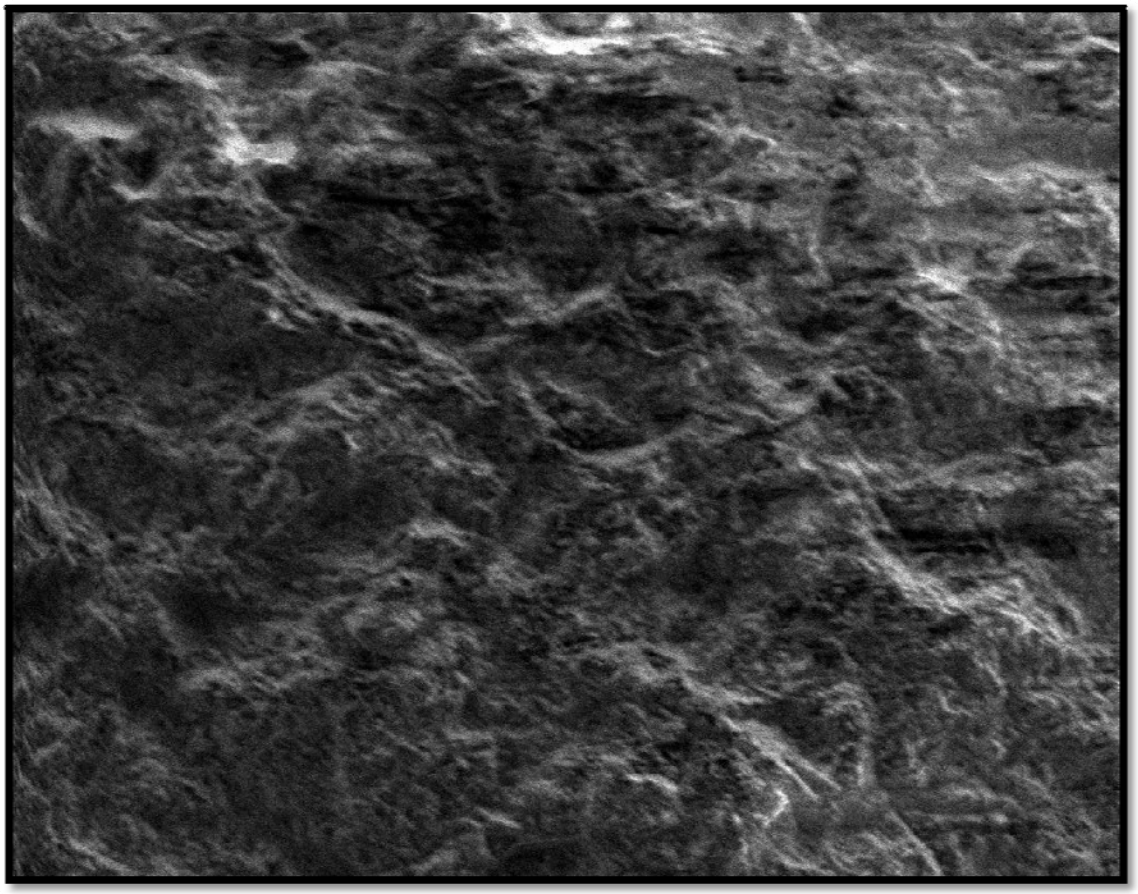

Figure 17. SEM micrograph (secondary electron mode at X750) of resin composite surface treated with airborne particle abrasion with 50-µm aluminum oxide particles.

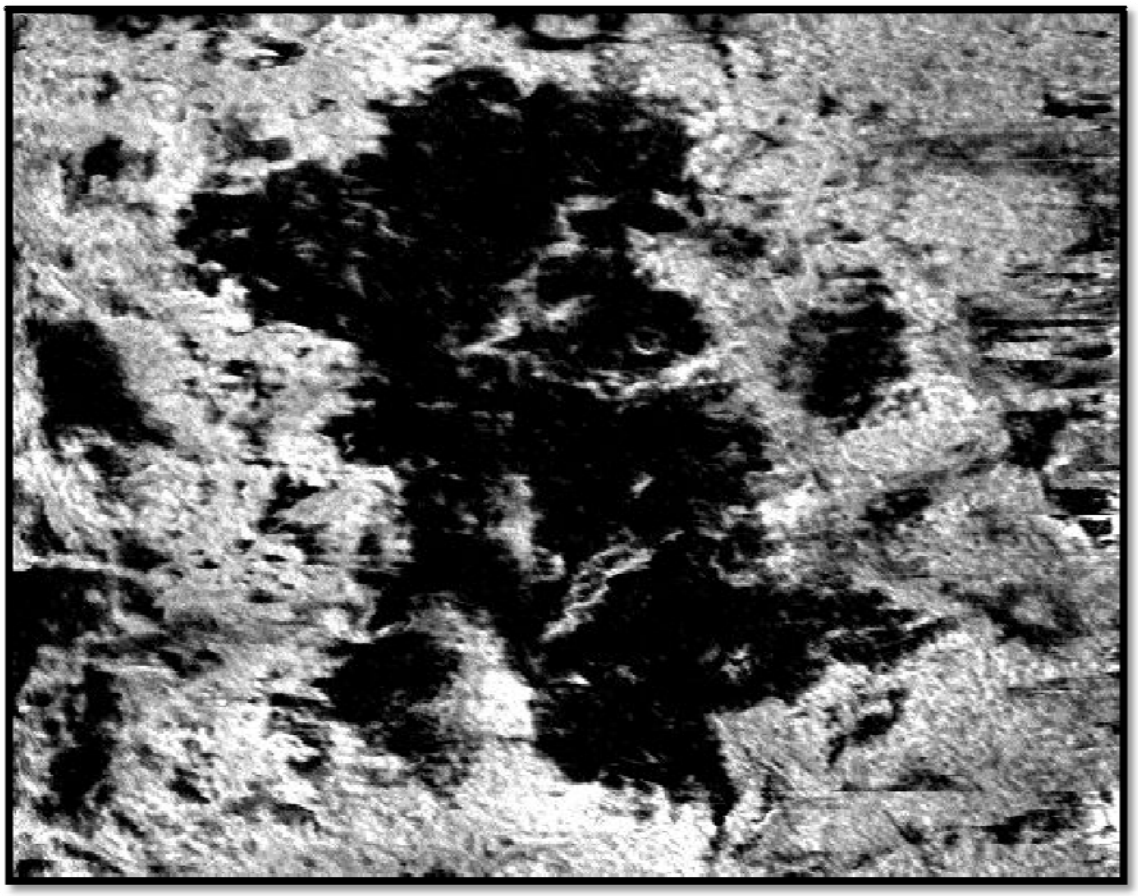

FIGURE 18. SEM micrograph (backscattered electron mode at X750) of resin composite surface treated with airborne particle abrasion with 50-µm aluminum oxide particles.

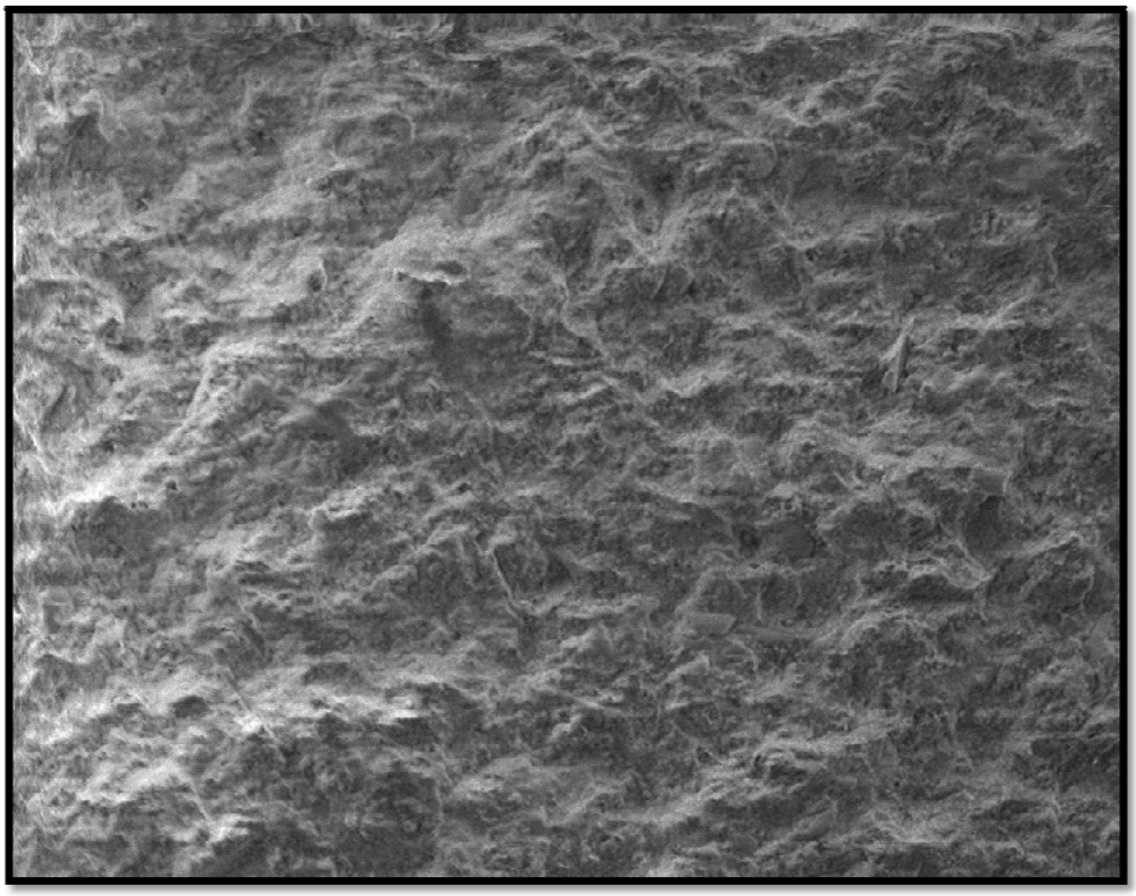

FIGURE 19. SEM micrograph (secondary electron mode at X750) of resin composite surface treated with tribochemical silica coating.

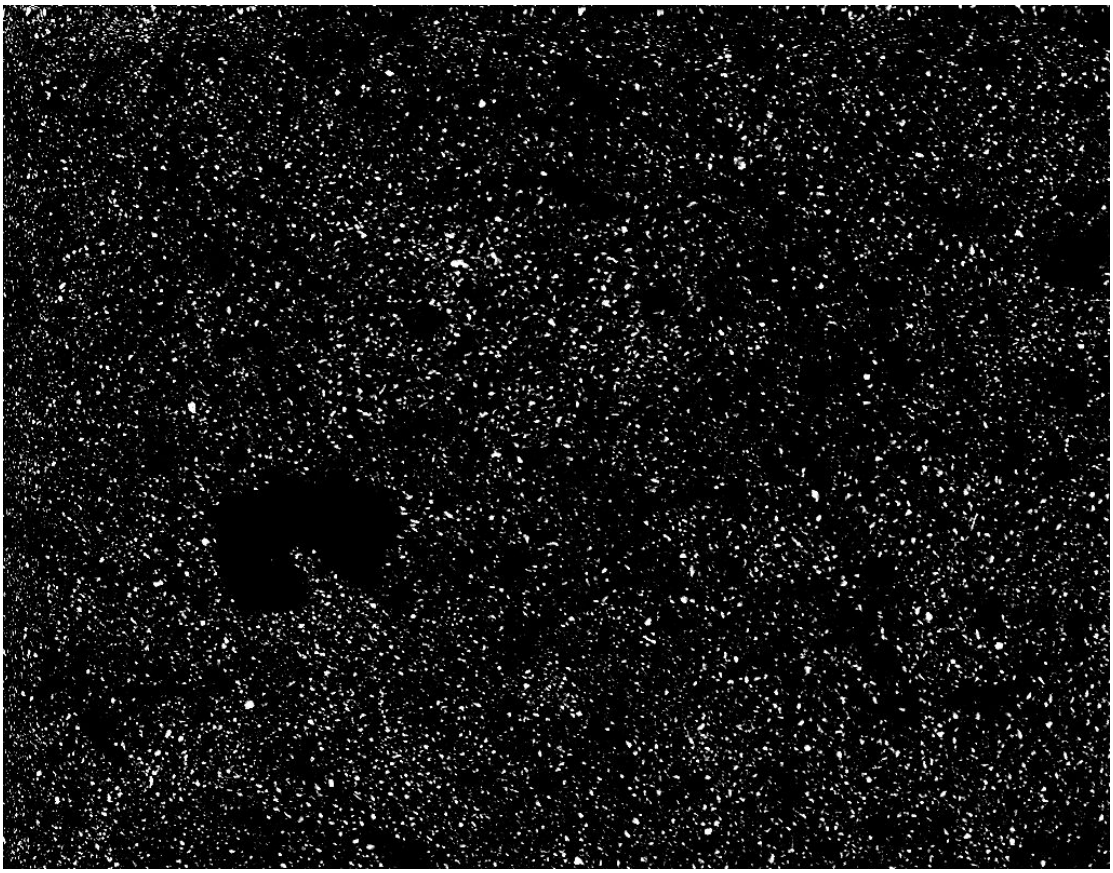

FIGURE 20. SEM micrograph (backscattered electron mode at X750) of resin composite surface treated with tribochemical silica coating.

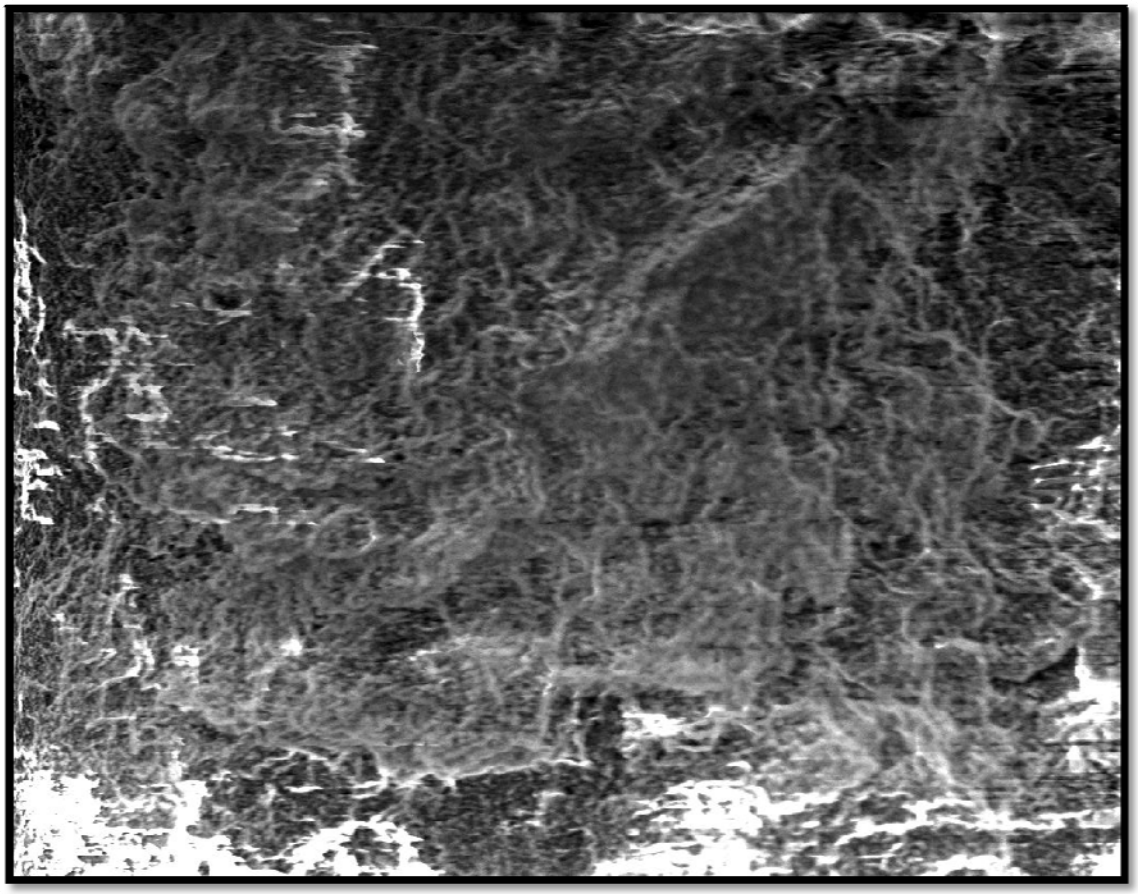

FIGURE 21. SEM micrograph (secondary electron mode at X750) of resin composite surface treated with Er,Cr:YSGG laser.

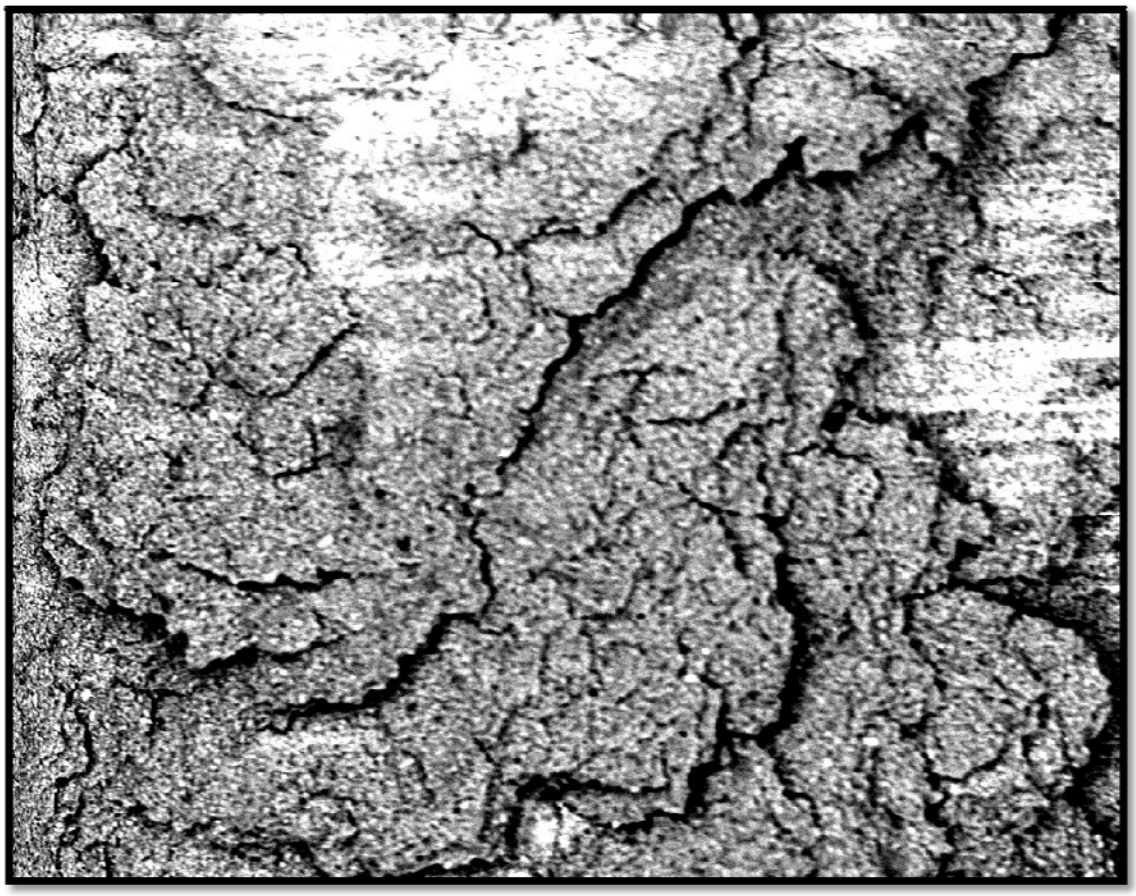

FIGURE 22. SEM micrograph (backscattered electron mode at X750) of resin composite surface treated with Er,Cr:YSGG laser.

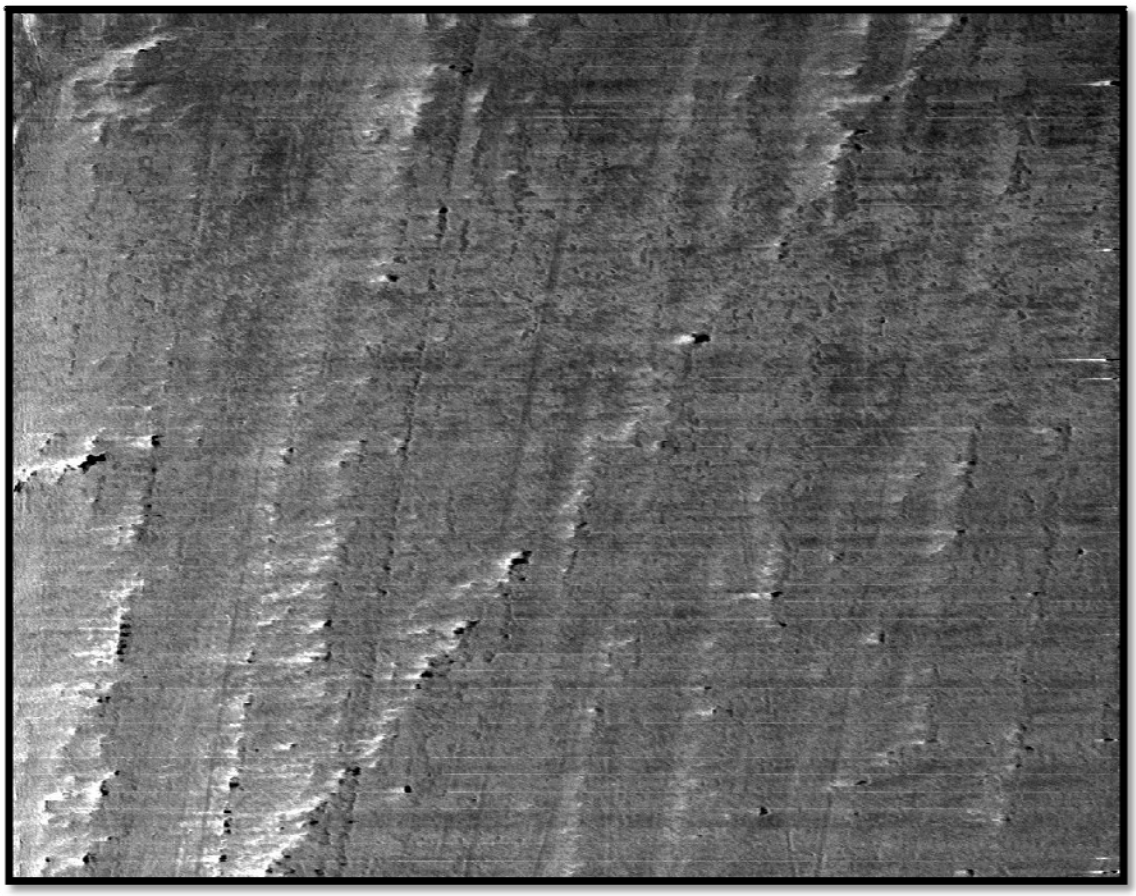

FIGURE 23. SEM micrograph (secondary electron mode at X750) of resin composite surface without suface treatment.

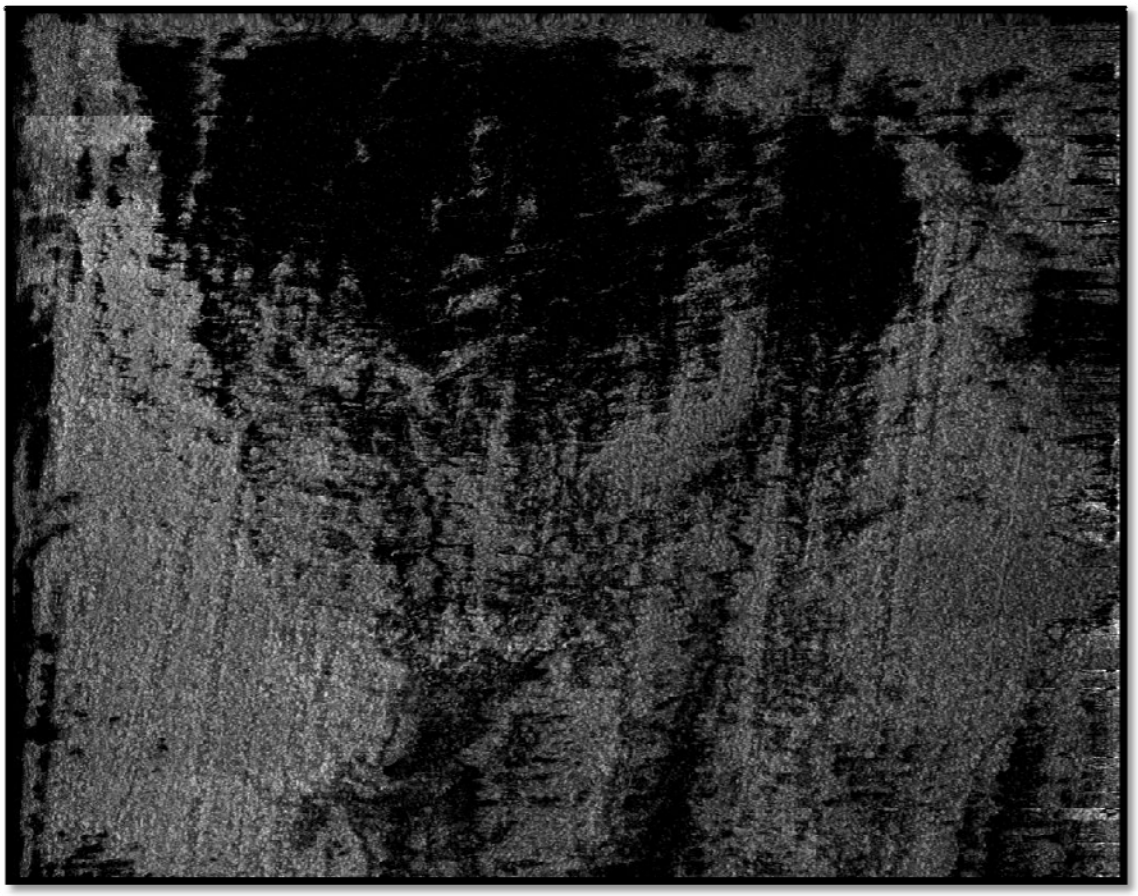

FIGURE 24. SEM micrograph (backscattered electron mode at X750) of resin composite surface without suface treatment.

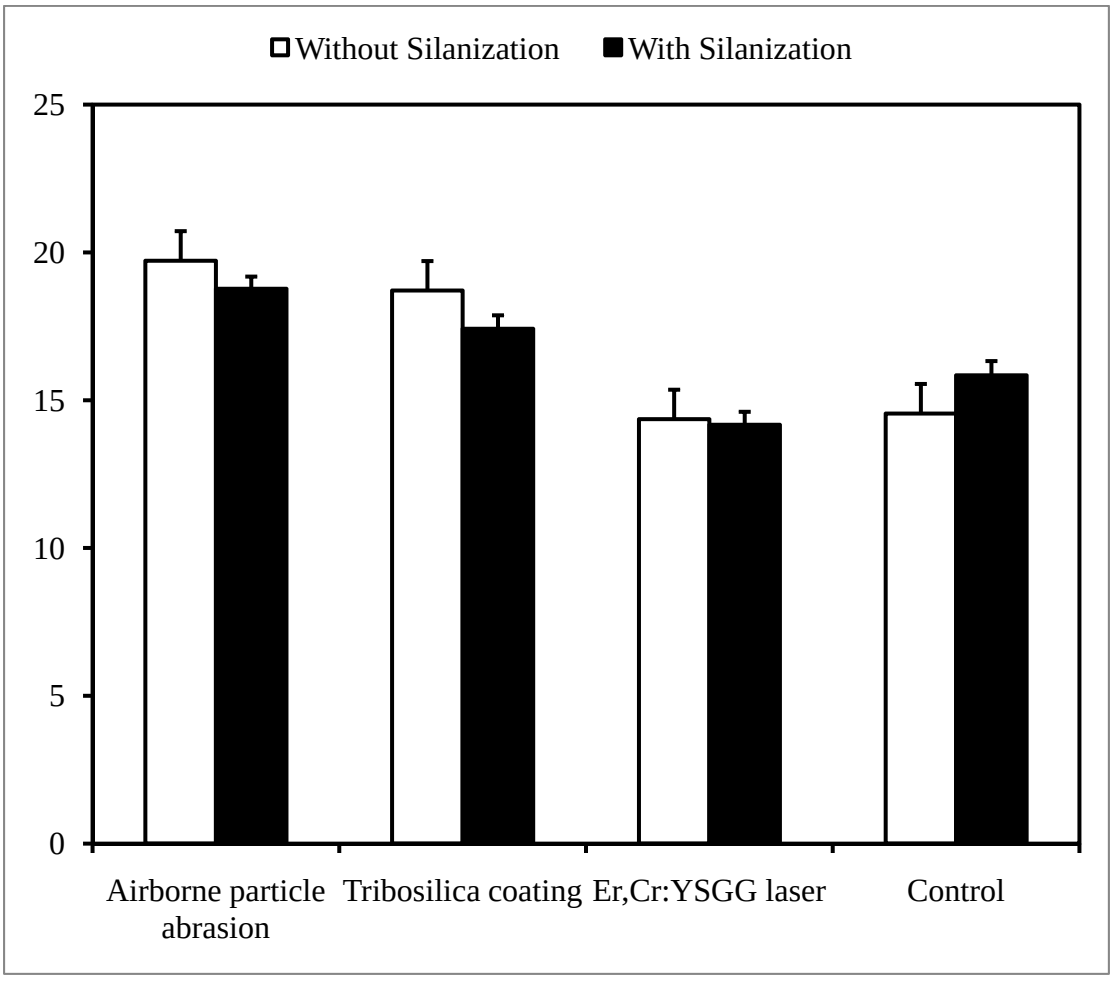

FIGURE 25. Bar graph showing repair bond strength (MPa) results.

DISCUSSION

This study was done in order to examine the effect of surface conditioning methods on repair bond strength of fresh to aged resin composite. Shear bond strength was tested because it provided an appropriate measurement of the maximum stress applied at the bonding interface between layers of new and old layer of resin composite. This study would be most useful in predicting the success of repairs in the anterior region, where restorations are commonly subjected to shear forces during function.

Many studies compared repair bond strength with the cohesive strength of intact resin composite,[17, 28, 33, 36, 38] whereas others set the control as no surface treatment before repairing to compare with other test groups.[23, 24, 28, 40, 44, 47, 62] However, the bond strength necessary for repairing resin composite restorations *in vivo* has not yet been defined. One measure, which might be comparable, is the bond strength between resin and etched enamel. The range of bond strength between resin-to-etched-enamel was reported to be from 15 to 30 MPa.[14, 32, 46] Resin composites seldom fail mechanically at the etched enamel interface, so the assumption could be made that an equivalent repair bond strength would be clinically adequate. However, since many factors affect bond strength, it is impossible to assume that the results of *in vitro* studies will accurately replicate the clinical situation. One can only say that the highest repair bond strength is still the goal.

The results of this study showed that airborne particle abrasion with 50 μm aluminum oxide particles without silane had the repair bond strength of 19.7 +/- 1.1 MPa and was significantly different from both Er,Cr:YSGG laser, and control. Bond strength in the tribochemical silica coating group was not different from the air abrasion group,

but significantly greater than the Er,Cr:YSGG laser and the non silanization control group. There was no significant difference in shear bond strength between the Er,Cr:YSGG laser group and the control group. Also, silanization did not produce any significant difference (Figure 25).

SURFACE TREATMENT

In this study, airborne particle abrasion with 50 μm aluminum oxide particles with the use of an intermediate material significantly improved the repair bond strength of the specimens compared to control. This result is in agreement with the majority of studies in this area.[20, 25, 26, 30-38] However, Bonstein et al.[24] found no difference when using airborne particle abrasion with 30 μm aluminum oxide particles compared to a control group which used only bonding agent. This improvement in bond strength could be due to the fact that airborne particle abrasion with aluminum oxide particles creates a rough (Table III), irregular surface with large micro-retentive areas (Figure 17) and increases the wettability for adhesive agent.

Surface treatment with tribochemical silica coating with 30 μm aluminum oxide particles coated with silica in this study also significantly improved repair bond strength compared to the control group. This result is supported by numerous studies that found this technique effective.[29, 35, 40-42, 63] In comparison to airborne particle abrasion with 50 μm aluminum oxide particles, Bouschlicher et al.[29] found that tribochemical silica coating was superior, whereas Pontes et al.[35] found no significant difference between the two techniques. It was concluded that the effectiveness of this technique comes from the ability to create micro-irregularities, increase surface energy, and embed a proprietary silicate ceramic layer. Surface roughness value and SEM characteristic of this surface

treatment revealed less irregular surface when compared to airborne particle abrasion with 50 μm aluminum oxide particles (Table III, Figure 19). It is expected that this technique should be more effective when used with a silane coupling agent to promote chemical interaction of the ceramic surface to a resin bonding agent. However, Rathke et al.,[27] found that tribochemical silica coating was inferior due to the smaller particle size and shallower abrasion of 30 μm particles compared to 50 μm. Our results showed that airborne particle abrasion with 50 μm aluminum oxide particles had similar effect on bond strength. The use of a silane coupling agent along with a tribochemical silica coating did not show any significant difference.

Bond strength of Er,Cr:YSGG abraded groups (group 3: 14.4 +/-1.5 MPa; group 7: 14.2 +/-1.4 MPa) did not show any improvement when compared to the control (group 4: 14.6 +/- 1.1 MPa; group 8: 15.8 +/- 1.5 MPa). Moreover, more adhesive failure was noticed in both laser treated groups. Surface roughness value and SEM characteristic revealed macro-irregularities and cracked lines created on resin composite surface (Table III, Figures 21 and 22). The result in this study is in contrast with Burnett et al.[61] who used Er:YAG (wavelength 2.94 μm) for treating indirect resin composites and found that it was superior to using fluoridic acid and airborne particle abrasion. In this study, a different wavelength of erbium laser was used (Er,Cr:YSGG 2.78 μm), as well as a different type of resin composite. It can be hypothesized that:

1) Er: YAG with a longer wavelength (2.94μm) produces more irregularities in the resin composite surface than a wavelength 2.78 μm.
2) Indirect resin composite has some components that absorb laser energy more readily than direct resin composite.

3) Burnett et al.[61] suggested that following irradiation with laser, the restoration should be cleaned in an ultrasonic bath for 2 minutes to remove the exploded particles. In this study, the specimens were only cleaned with phosphoric acid. That might have been insufficient to remove any interference.

SILANIZATION

Silanization in this study did not significantly improved repair bond strength for any surface treatment groups on control specimens. Brosh et al.[26] also found that using silane coupling agent showed no significant improvement of bond strength when comparing surface conditions without adhesive and silane, with adhesive, and with adhesive and silane. In addition, some studies found lower repair bond strengths when using a silane coupling agent after using bur[10] and air abrasion.[38] Bonstein et al.[24] proposed that silane may change the structure of the matrix of aged resin composite and leave the filler particles bare, leading to a reduction in retention. However, Cesar et al.[30] showed higher repair bond strength when using silane following air abrasion and Hisamatsu et al.[43] demonstrated the effectiveness of using silane primer and bonding agent on microfilled resin composite after surfacing specimens with abrasive paper. Bouschlicher et al.[29] showed that the use of silane coupling agent on hybrid resin composite had significantly greater repair bond strength after bur and air abrasion. Fawzy et al.[28] demonstrated that using silane primer before the application of adhesive significantly improved repair bond strength due to the fact that the silane primer is capable of forming chemical bonds with smeared grinding debris. Based on these results, the effect of silane coupling agent is still controversial.

BONDING AGENT

Bonding agent has been routinely used as an intermediate adhesive when restoring teeth with resin composite. In resin repair, application of bonding agent is felt to enhance the composite-composite bond by promoting chemical coupling to the resin matrix and mechanical retention through penetration into the retentive areas in the matrix. In the present study, bonding agent was used in all groups including the two control groups to simulate the clinical situation. Recently, Rathke et al.[27] showed that adhesive treatment following mechanical treatment significantly increased the bond strength of 6-month-old resin composite when compared to mechanical treatment alone. This is in agreement with the majority of studies.[17, 23, 26, 33, 43, 47] However, a few studies found no significant difference. [25, 28]

AGING PROCESS

The purpose for taking all specimens through an aging process is to simulate the oral condition. However, there are many factors in the oral condition (temperature, pH, water sorption) that are difficult to replicate in an *in vitro* experiment. In this study, thermocycling was chosen as the aging method to attempt some exposure to both thermal stress and water sorption as well as elimination of some unreacted carbon-carbon double bond. According to Gale et al.[64, 65] the cycle of thermal change in the oral situation could be occurring 20 to 50 times a day, or approximately 10,000 cycles a year. If this is true, then thermocycling 5000 times might represent 6 months in service. Ozcan et al.[63] found that the water sorption effect of thermocycling conditions has a deleterious effect on repair strength due to hydrolytic degradation that occurs by oxidation and/or hydrolysis process. Also, they suggested that temperature alterations could decrease the number of

unreacted double bonds on the resin surface. Based on their study, thermocycling represents a more challenging aging condition than immersion in citric acid and boiling in water for 8 hours. However, Bredeke et al.[42] found that aging through water storage for 2 months showed significantly worse repair bond strength than immersion in deionized water for 1 week, immersion in citric acid in 1 week, boiling in water 8 hours, and thermocycling 5000 times between 5°C and 55°C.

Based on these studies, it can be theorized that the cohesive failure in control groups might be caused by two factors. Firstly, insufficient aging from the thermocycling process which actually took approximately 52 hours for 5000 cycles. This may be enough to create thermal stress in the resin composite, but not enough to dissolve all unreacted carbon-carbon double bonds. Secondly, the bonding agent (Optibond Solo Plus, SDS Kerr) might be very effective in penetrating into micro-cracks in the surface and creating a strong covalent bond to the aged resin composite.

SUMMARY AND CONCLUSIONS

This *in vitro* study was conducted to evaluate which surface conditioning methods provides the greatest bond strength when repairing resin composite. It was also to evaluate the effectiveness of using a silane coupling agent with each surface treatment.

In this study, resin composite was placed into the center of a plastic mold which was filled with acrylic resin. Eighty specimens were used and aged by thermocycling 5000 times between 6°C and 51°C (+/- 2°C). A 30-second dwell time and 10-second transfer time were used. Specimens were randomly divided into four groups of 20 specimens each into airborne particle abrasion with 50 μm aluminum oxide particles, tribochemical silica coating, Er,Cr:YSGG laser, and control. Then, the four groups were further divided into two subgroups, one was silanated, another was not. Airborne particle abrasion groups were done by treating the surface with 50 μm aluminum oxide particles for 10 seconds at a pressure of 60 psi, and 5 mm distance from tip of microetcher unit to specimen surface. Tribochemical silica coating groups were done by treating the surface with 30 μm aluminum oxide particles coated with silica for 10 seconds at the same pressure and distance as the previous groups. Er,Cr:YSGG laser groups were done by treating the surface with laser machine in noncontact and focused mode, with a cylinder fiber tip moving around the specimen surface at a distance of approximately 1 mm for 10 seconds. The laser parameters were set at a wavelength of 2.78 μm, a pulse frequency of 20 Hz, a pulse duration of 140 μs, a power of 4.0 w, an air pressure setting of 65 percent, and a water pressure setting of 55 percent. There was no treatment for control groups. All specimens were cleaned with 35 percent phosphoric acid, rinsed, and air dried. In the

silane treated groups, a silane coupling agent was applied for 60 seconds, then the specimens were air dried. An adhesive agent was applied, the solvent was evaporated using canned compressed air, and the specimens were light-cured for 20 seconds. A new resin composite layer was applied into the plastic mold diameter 4 mm, and 2 mm high. All specimens were stored in distilled water at 37°C for 24 hours. A shear test was performed with a Universal mechanical testing machine. Failure load and type of failure were recorded and analyzed with two-way ANOVA. In the results of this study, airborne particle abrasion with 50-µm aluminum oxide particles and tribochemical silica coating groups had significantly higher repair bond strength than Er,Cr:YSGG laser and control groups. There was no significant difference between abrasion with 50 µm aluminum oxide particles and tribochemical silica coating groups. Also, there was no significant difference between Er,Cr:YSGG laser and control groups. Silanization did not show any significance for repair bond strength.

It was concluded that:

1) The use of airborne particle abrasion and tribochemical silica coating in combination with a bonding agent significantly increased the repair bond strength.
2) Surface treatment with Er,Cr:YSGG did not show any improvement in bond strength.
3) Silanization did not improve the repair bond strength.

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APPENDIXES

APPENDIX I

Group 1 data

Specimen#	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	4.053	252.401	19.6	12.9016	127.571
2	4.073	256.193	19.7	13.0292	124.688
3	4.057	228.467	17.7	12.9271	108.673
4	4.080	259.924	19.9	13.0741	130.614
5	4.047	244.737	19.0	12.8634	118.202
6	4.047	246.454	19.2	12.8634	116.615
7	4.043	247.335	19.3	12.8380	128.067
8	4.020	276.011	21.7	12.6923	153.351
9	4.087	263.285	20.1	13.1190	137.649
10	4.050	270.885	21.0	12.8825	150.777
Mean	4.056	254.569	19.7	12.9191	129.621
Std. Dev.	0.020	13.881	1.1	0.1256	14.320
% COV	0.49	5.45	5.66	0.97	11.05
Minimum	4.020	228.467	17.7	12.6923	108.673
Maximum	4.087	276.011	21.7	13.1190	153.351

APPENDIX II

Group 2 data

Specimen#	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	4.060	238.235	18.4	12.9462	120.625
2	4.043	229.973	17.9	12.8380	100.383
3	4.013	240.842	19.0	12.6482	109.658
4	4.043	256.216	20.0	12.8380	139.593
5	4.047	220.422	17.1	12.8634	90.146
6	4.057	272.871	21.1	12.9271	142.474
7	4.080	254.617	19.5	13.0741	126.110
8	4.050	243.185	18.9	12.8825	123.440
9	4.043	219.376	17.1	12.8380	93.112
10	4.057	233.495	18.1	12.9271	110.790
Mean	4.049	240.923	18.7	12.8782	115.633
Std. Dev.	0.017	16.714	1.3	0.1084	18.070
% COV	0.42	6.94	6.74	0.84	15.63
Minimum	4.013	219.376	17.1	12.6482	90.146
Maximum	4.080	272.871	21.1	13.0741	142.474

APPENDIX III

Group 3 data

Specimen #	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	3.990	194.592	15.6	12.5036	88.577
2	4.040	198.955	15.5	12.8190	87.985
3	4.020	194.732	15.3	12.6923	75.980
4	4.080	187.817	14.4	13.0741	75.942
5	4.040	201.123	15.7	12.8190	90.231
6	4.050	180.476	14.0	12.8825	77.195
7	4.110	185.452	14.0	13.2670	73.541
8	4.070	183.973	14.1	13.0100	73.896
9	4.060	136.001	10.5	12.9462	37.998
10	4.060	187.630	14.5	12.9462	73.195
Mean	4.052	185.075	14.4	12.8960	75.454
Std. Dev.	0.033	18.496	1.5	0.2095	14.777
% COV	0.81	9.99	10.57	1.62	19.58
Minimum	3.990	136.001	10.5	12.5036	37.998
Maximum	4.110	201.123	15.7	13.2670	90.231

APPENDIX IV

Group 4 data

Specimen#	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	4.100	185.521	14.1	13.2025	80.083
2	4.240	201.592	14.3	14.1196	90.231
3	4.200	184.686	13.3	13.8544	66.399
4	4.070	208.425	16.0	13.0100	83.544
5	4.060	208.393	16.1	12.9462	92.714
6	4.130	175.757	13.1	13.3965	68.258
7	4.080	175.519	13.4	13.0741	68.202
8	4.120	202.907	15.2	13.3317	88.815
9	4.110	199.766	15.1	13.2670	82.834
10	4.090	196.258	14.9	13.1382	80.223
Mean	4.120	193.882	14.6	13.3340	80.130
Std. Dev.	0.058	12.576	1.1	0.3764	9.583
% COV	1.40	6.49	7.43	2.82	11.96
Minimum	4.060	175.519	13.1	12.9462	66.399
Maximum	4.240	208.425	16.1	14.1196	92.714

APPENDIX V

Group 5 data

Specimen#	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	4.053	232.874	18.1	12.9016	104.261
2	4.040	212.051	16.5	12.8190	85.292
3	4.050	236.837	18.4	12.8825	112.890
4	4.057	254.024	19.7	12.9271	129.323
5	4.013	246.921	19.5	12.6482	118.101
6	4.047	266.371	20.7	12.8634	137.229
7	4.047	226.249	17.6	12.8634	103.459
8	4.013	257.843	20.4	12.6482	124.968
9	4.057	233.820	18.1	12.9271	98.843
10	4.057	241.670	18.7	12.9271	115.248
Mean	4.043	240.866	18.8	12.8407	112.962
Std. Dev.	0.017	16.065	1.3	0.1072	15.504
% COV	0.42	6.67	6.93	0.83	13.73
Minimum	4.013	212.051	16.5	12.6482	85.292
Maximum	4.057	266.371	20.7	12.9271	137.229

APPENDIX VI

Group 6 data

Specimen#	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	4.037	233.277	18.2	12.7999	112.796
2	4.027	224.413	17.6	12.7366	94.854
3	4.097	237.602	18.0	13.1832	109.689
4	4.107	210.212	15.9	13.2477	88.654
5	4.053	202.148	15.7	12.9016	80.652
6	4.060	210.780	16.3	12.9462	85.754
7	4.083	269.521	20.6	13.0933	149.133
8	4.060	231.573	17.9	12.9462	112.820
9	4.050	223.577	17.4	12.8825	99.609
10	4.050	213.465	16.6	12.8825	94.665
Mean	4.062	225.657	17.4	12.9620	102.863
Std. Dev.	0.026	19.226	1.4	0.1639	19.763
% COV	0.63	8.52	8.30	1.26	19.21
Minimum	4.027	202.148	15.7	12.7366	80.652
Maximum	4.107	269.521	20.6	13.2477	149.133

APPENDIX VII

Group 7 data

Specimen#	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	4.080	191.185	14.6	13.0741	70.247
2	4.050	202.239	15.7	12.8825	75.823
3	4.100	198.305	15.0	13.2025	77.571
4	3.990	173.628	13.9	12.5036	57.377
5	4.070	165.072	12.7	13.0100	53.515
6	4.090	178.503	13.6	13.1382	61.744
7	4.130	219.510	16.4	13.3965	85.195
8	4.080	192.928	14.8	13.0741	65.204
9	4.080	155.913	11.9	13.0741	47.863
10	4.090	171.662	13.1	13.1382	60.615
Mean	4.076	184.895	14.2	13.0494	65.515
Std. Dev.	0.037	19.318	1.4	0.2330	11.650
% COV	0.90	10.45	9.81	1.79	17.78
Minimum	3.990	155.913	11.9	12.5036	47.863
Maximum	4.130	219.510	16.4	13.3965	85.195

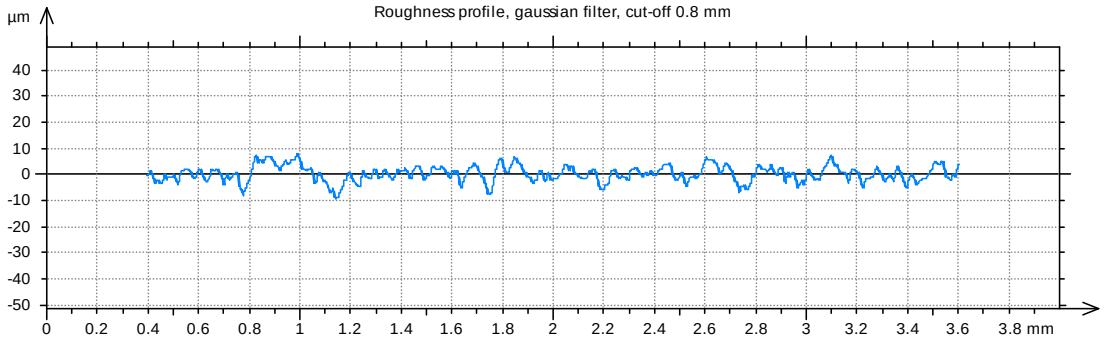
APPENDIX VIII

Group 8 data

Specimen#	Diameter Mm	Peak Load N	Peak Stress MPa	Area mm^2	Energy to Break N*mm
1	4.090	224.951	17.1	13.1382	100.746
2	4.090	214.166	16.3	13.1382	96.934
3	4.080	206.032	15.8	13.0741	80.228
4	4.090	204.573	15.6	13.1382	83.860
5	4.080	217.990	16.7	13.0741	100.072
6	4.190	178.399	12.9	13.7885	58.063
7	4.280	210.782	14.7	14.3872	87.887
8	4.070	195.267	15.0	13.0100	85.939
9	4.070	242.933	18.7	13.0100	119.184
10	4.100	205.840	15.6	13.2025	94.033
Mean	4.114	210.093	15.8	13.2961	90.695
Std. Dev.	0.068	17.218	1.5	0.4446	15.994
% COV	1.65	8.20	9.71	3.34	17.64
Minimum	4.070	178.399	12.9	13.0100	58.063
Maximum	4.280	242.933	18.7	14.3872	119.184

APPENDIX IX

Roughness for airborne particle abrasion with 50-µm Al_2O_3 Specimen #1*

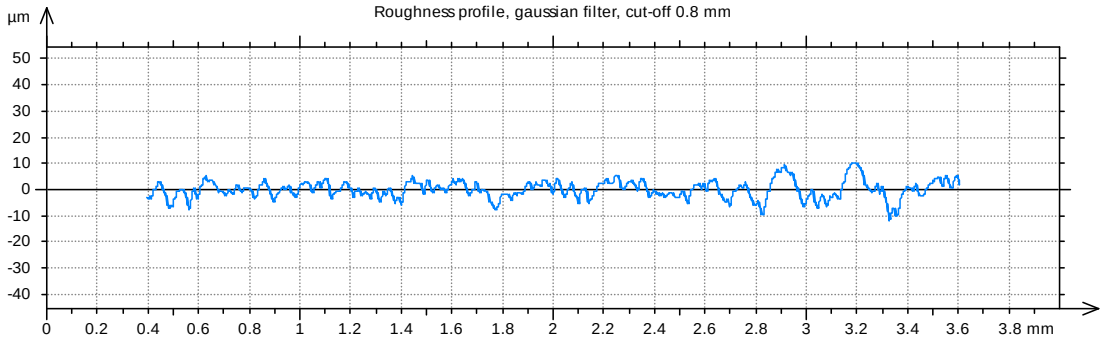


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 2.44 µm

APPENDIX X

Roughness for airborne particle abrasion with 50-µm Al_2O_3 Specimen #2*

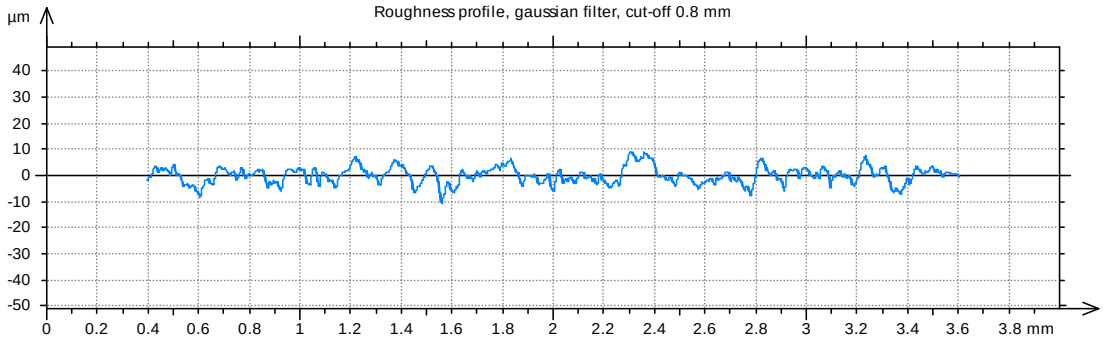


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 2.12 µm

APPENDIX XI

Roughness for airborne particle abrasion with 50-µm Al_2O_3 Specimen #3*

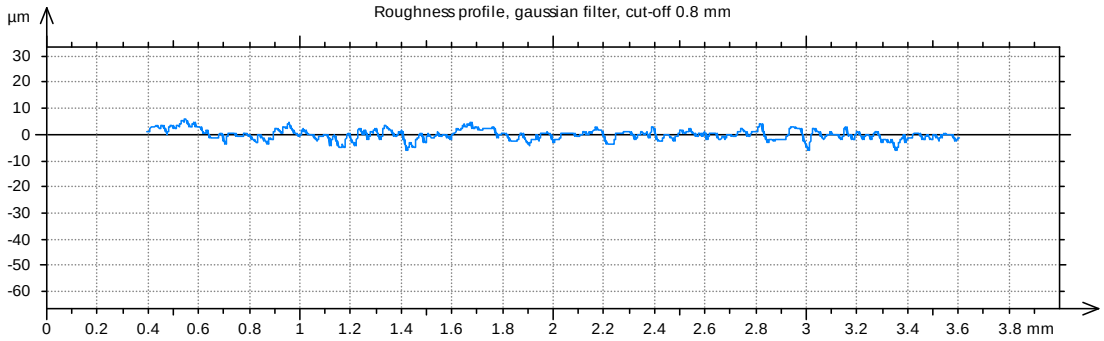


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 2.55 µm

APPENDIX XII

Roughness for tribosilica coating Specimen #1*

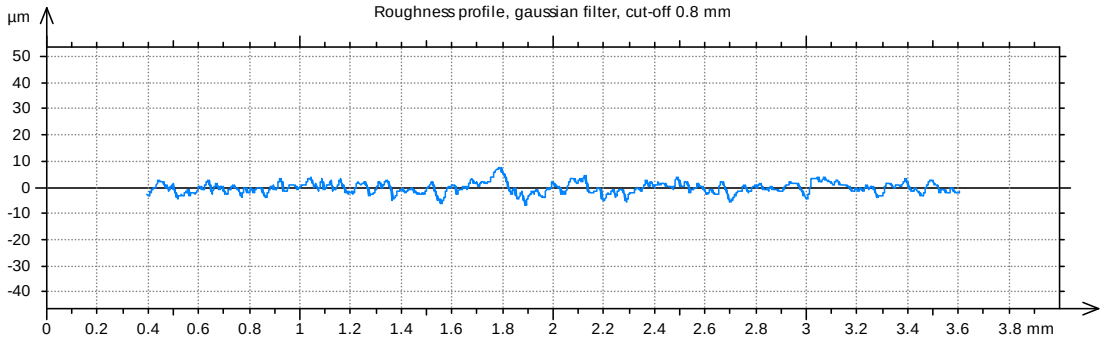


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 1.55 µm

APPENDIX XIII

Roughness for tribosilica coating Specimen #2*

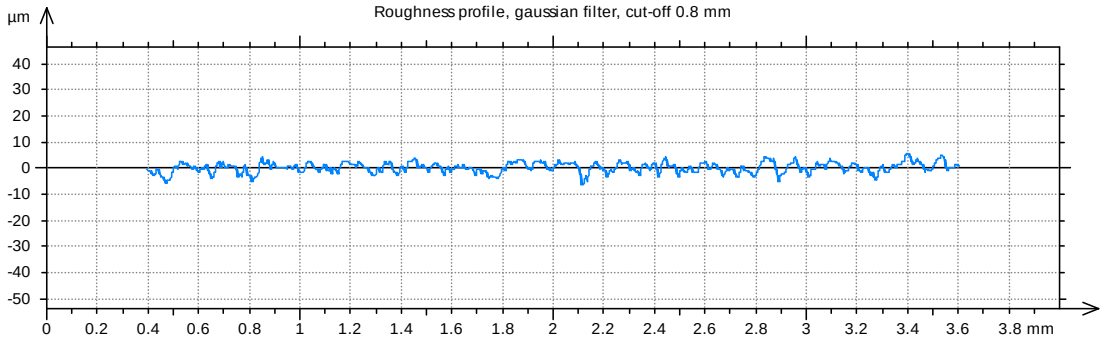


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 1.72 µm

APPENDIX XIV

Roughness for tribosilica coating Specimen #3*

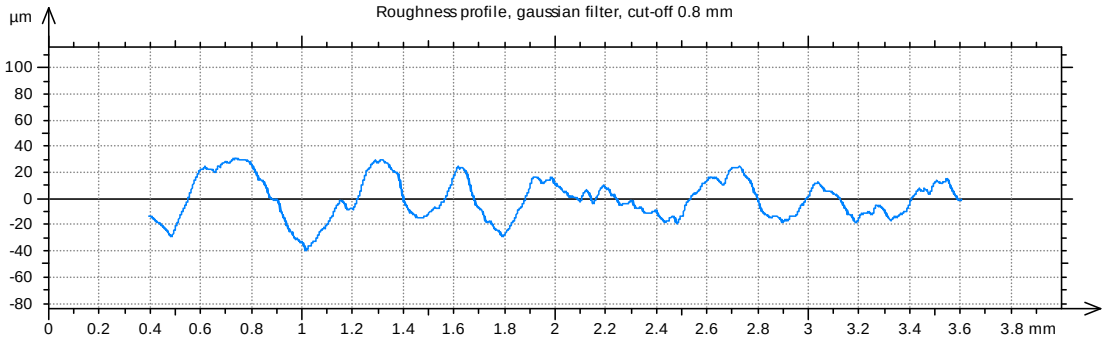


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 1.5 µm

APPENDIX XV

Roughness for Er,Cr:YSGG laser Specimen #1*

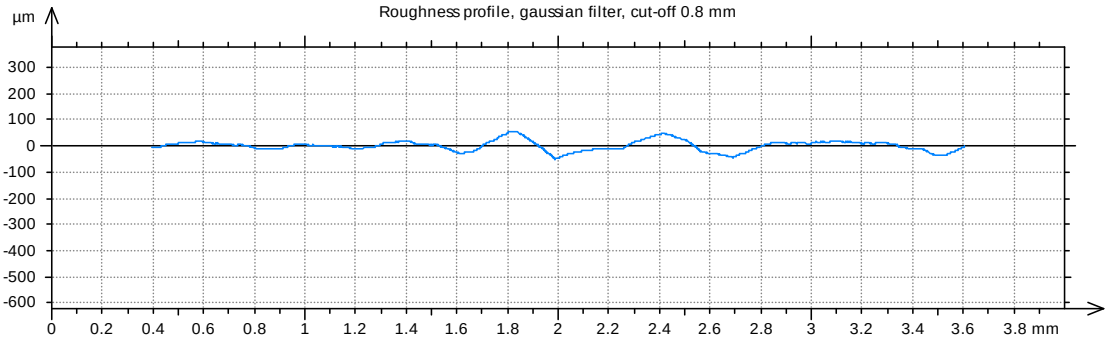


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 14.4 µm

APPENDIX XVI

Roughness for Er,Cr:YSGG laser Specimen #2*

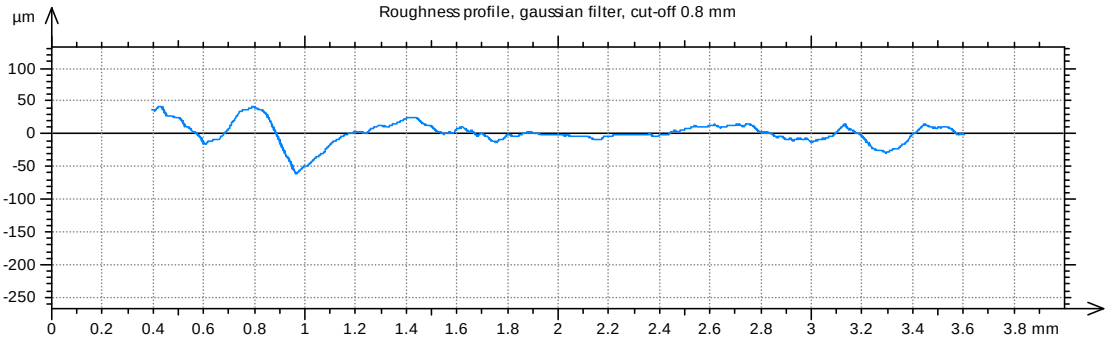


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 16.4 µm

APPENDIX XVII

Roughness for Er,Cr:YSGG laser Specimen #3*

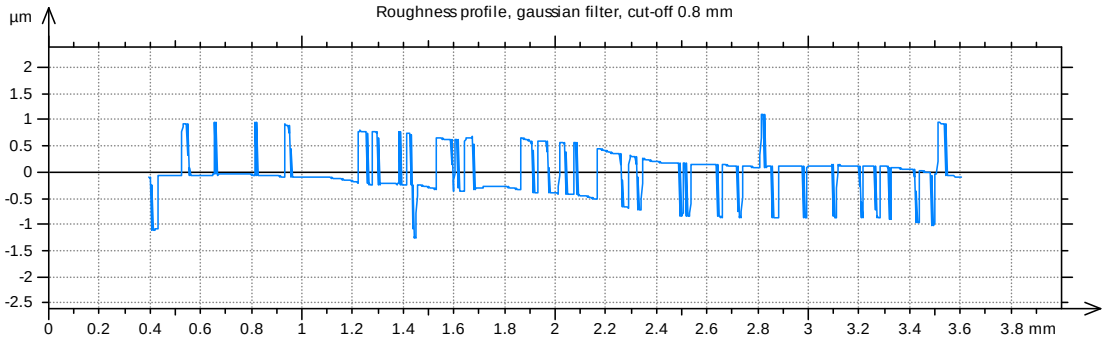


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 12.3 µm

APPENDIX XVIII

Roughness for control Specimen #1*

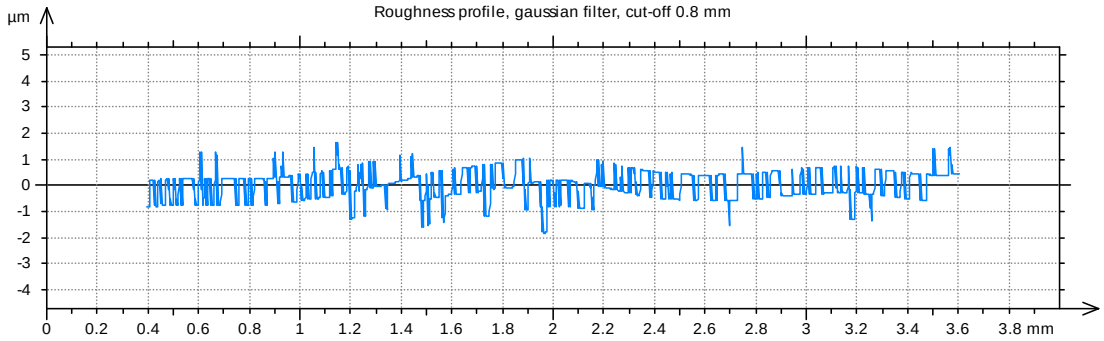


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 0.308 µm

APPENDIX XIX

Roughness for control Specimen #2*

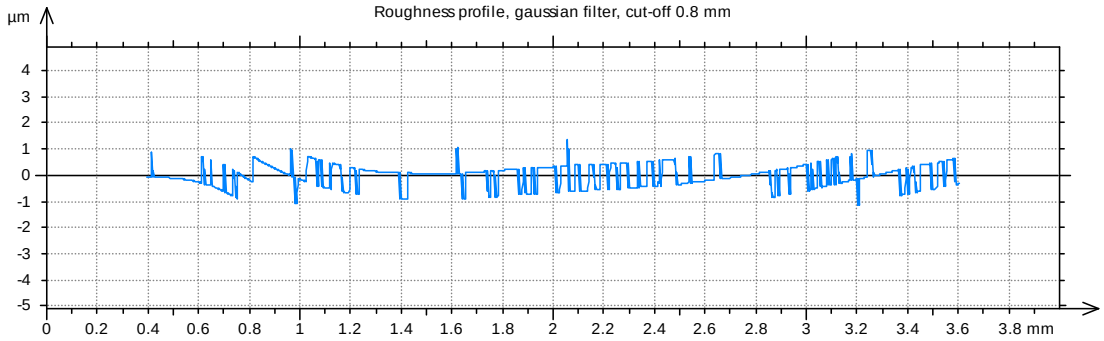


Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 0.461 µm

APPENDIX XX

Roughness for control Specimen #3*



Parameters calculated on the profile

*Parameters calculated by means of all the sampling lengths. 1) Microroughness filtering ratio = 2.5 µm. 2) Gaussian filter = 0.8 mm. 3) Ra = 0.321 µm

ABSTRACT

EFFECT OF SURFACE CONDITIONING METHODS ON REPAIR BOND STRENGTH OF MICROHYBRID RESIN MATRIX COMPOSITE

by

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Repair is an alternative treatment option in many cases to replacement of resin matrix composite restoration. However, aged resin matrix composites have a limited number of carbon-carbon double bonds to adhere to a new layer of rein. Therefore, surface treatments of the aged resin matrix composite surface prior to repairing could improve the repair bond strength.

The objectives of this study were to: 1) To evaluate various surface treatments on shear bond strength of repair between aged and new microhybrid resin matrix composite,

and 2) To assess the influence of applying a silane coupling agent after surface treatments.

Eighty disk-shaped resin matrix composite specimens were fabricated and thermocycled 5000 times prior to surface treatment. Specimens were randomly assigned to one of the three surface treatments (n = 20): 1) Airborne abrasion with 50 μm aluminum oxide, 2) Tribochemical silica coating (CoJet), or 3) Er,Cr:YSGG laser and control group (n = 20). Specimens were cleaned with 35-percent phosphoric acid, rinsed, and dried. Each group was assigned into two subgroups (n =10): a) no silanization, and b) with silanization. Adhesive agent was applied and new resin matrix composite was bonded to each conditioned surface. Bond strength was evaluated by shear test. Data were analyzed with a two-way ANOVA model.

The interaction between conditioning and silanization was significant ($p = 0.0163$), indicating that comparisons of silanization must be evaluated for each conditioning method, and that comparisons of conditioning methods must be evaluated separately with and without silanization. Airborne particle abrasion showed significantly higher repair bond strength than Er,Cr:YSGG laser without silanization ($p < 0.0001$) and with silanization($p = 0.0002$), and higher repair bond strength than the control without silanization ($p < 0.00001$) and with silanization ($p < 0.00001$). Airborne particle abrasion did not have significantly different in repair bond strength than Tribosilica coating without silanization ($p = 0.70$) or with silanization ($p = 0.33$). Tribosilica coating had significantly higher repair bond strength than Er,CR:YSGG laser without silanization ($p < 0.0001$) and with silanization ($p < 0.0001$), and significantly higher repair bond strength than control without silanization ($p < 0.0001$), but not with silanization ($p =$

0.16). Er,CR:YSGG laser and control did not have significantly different repair bond strength without silanization ($p = 1.00$) or with silanization ($p = 0.11$). There was no effect of silanization on repair bond strength overall ($p = 0.34$) for any of the surface conditioning methods ($p = 0.76$ for airborne particle abrasion; $p = 0.39$ for tribosilica coating; $p = 1.00$ for Er,Cr:YSGG laser, or $p = 0.39$ for control).

Airborne particle abrasion with 50-µm aluminum oxide particle and tribochemical silica coating followed by the application of bonding agent provided the highest shear bond strength values, suggesting that they might be adequate methods to improve the quality of the repairs of resin-matrix composites.

CURRICULUM VITAE

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